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EXPLORATORY NONDESTRUCTIVE EVALUATION (NDE) RESEARCH FOR ADVANCED MATERIALS AND PROCESSES AND AGING SYSTEMS

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13. ABSTRACT (Maximum 200 words)

A sensor has been developed and tested that is capable of emitting and receiving ultrasonic energy at temperatures exceeding 900 C (1652 F) and pressures above 150 MPa (22,500 psi). The sensor works with standard ultrasonic pulser-receivers and has demonstrated the capability of measuring workpiece deformation during hot isostatic pressing (HIP). Details of the sensor design, performance, and coupling of the ultrasound to the workpiece are described. Ultrasonic data acquired by the sensor, *in situ*, during HIP runs are presented.

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FORWARD

The research work described in this final report was conducted under a Broad Agency Announcement program for Exploratory Nondestructive Evaluation (NDE) Research for Advanced Materials and Processes and Aging Systems. One of the purposes of the program was to identify and validate "novel approaches which will result in new NDE/I capabilities, or improvements of existing technologies which show promise in advancing the reliability, sensitivity, affordability or speed of NDE/I for Air Force applications." For novel approaches, the laboratory experiments were to be conducted using simplified specimens to verify the physical or chemical principles upon which the approach is based. The University of Dayton Research Institute conducted research to demonstrate the feasibility of using a special form of aluminum nitride in the development of a high temperature, ultrasonic sensor to measure deformation of work pieces in hot isostatic pressing vessels. The conditions inside hot isostatic pressing (HIP) vessels typically exceed 900 °C (1652 °F) and pressures exceed 100 MPa (14,500 psi). Very few sensing devices can tolerate these severe conditions. Demonstration of an ultrasonic sensor that would operate *in situ* during HIP runs would result in the potential for reduced HIP costs and time. Additionally, the increased process control could result in improved materials as the adverse effects of heat treatment on some materials could be minimized.

The research conducted during this program resulted in a working prototype sensor capable of emitting and receiving ultrasonic energy in a HIP vessel at temperatures greater than 900 °C (1652 °F) and pressures above 155 MPa (22,500 psi). Details of the sensor design and construction, coupling of the ultrasonic energy from the sensor to a work piece, and performance of the sensor inside a HIP vessel at typical operating temperature and pressure are described.

As is the case with most research projects, a team of engineers made the work possible. The members of the UDRI team were Robert J. Andrews, George A. Hartman, Dr. Prasanna Karpur, William J. Porter, Mark J. Ruddell, Norman D. Schehl, James D. Wolf, Larry D. Sqrow, and Richard A. Grant. Dr. Rollie Dutton with the National Institute of Standards and Technology (NIST) worked with the UDRI team as a consultant on the appropriate application of the sensor to actual HIP vessel environments. Conversations concerning the project's technical direction with Dr. Thomas J. Moran and Dr. Renee M. Kent (Wright Laboratories, Materials Directorate, Metals and Ceramics Division, NDE Branch) were appreciated. The preceding research by Dr. N.D. Patel of Fallon Ultrasonics, Inc. to develop the CVD process for the AlN film was important to the success of this project. The initial investigation of the high temperature performance of AlN material by UDRI was made possible by Dr. Lee Semiatin of the U.S. Air Force (Wright Laboratories, Materials Directorate, Metals and Ceramics Division, Materials Behavior Branch). Appreciation is expressed to Industrial Materials Technology (IMT), Inc. in London, Ohio, for the use of the HIP vessel, and Andrew Clow and Richard McHenry (with IMT) for assistance in conducting the HIP tests.

1. INTRODUCTION

At the present time, high temperature structural components, such as structural castings for jet engines, nickel-base superalloy turbine blades, and ceramic and metal matrix composites made from powders are often densified by hot isostatic pressing (HIP). However, currently available measurement devices (dilatometers) for monitoring the deformation of the HIP work piece are only useful on a few geometries and frequently fail during the HIP process. Thus, it is typical practice to use the maximum rated pressure of the vessel and to hold the work piece at the processing temperature for an extended time to be assured that the work piece is fully dense. Typical HIP times often exceed several hours to assure densification even though densification is complete in only a fraction of this time. The extra HIP time is inefficient in terms of cost and production runs and also can produce unwanted changes in the microstructure of the work piece.

The goal of the research described in this report was to demonstrate the feasibility of constructing an ultrasonic sensor to monitor, *in situ*, the deformation, density and/or microstructure of the HIP work piece during processing. The sensor was developed using a uniquely ordered form of aluminum nitride (AlN) created through a chemical vapor deposition technique [1]. The research succeeded in producing a sensor that operates in a typical HIP environment - temperatures above 900 °C (1652 °F) and pressures greater than 100 MPa (14,500 psi) - and demonstrated the feasibility of using the sensor to acquire data on the deformation of the work piece in the HIP vessel. The research has produced a sensor that is ready to be fully developed for use in production HIP vessels. Implementation of the sensor will allow the use of shorter HIP cycles, lower processing pressures and temperatures, and maximize the yield of acceptable products.

This report is organized in the same manner as was the contract statement of work (SOW). Each task of the SOW is included as italicized text at the beginning of each section followed by a detailed description of the research that led to the completion of the task. Appendix A contains a technical paper that will be published in the proceedings of the 1997 Annual Meeting of The Minerals, Metals, and Materials Society (TMS) in Orlando, Florida (Feb. 9-13, 1997, Session IV: *Applications of Sensors and Modeling to Materials Processing*). Appendix B contains a technical paper that will be published in the proceedings of the 1996 SPIE Symposium on Non-Destructive Evaluation for Aging Infrastructure and Manufacturing in Scottsdale, Arizona, December 2-4, 1996 (Session 2948: *Nondestructive Evaluation for Process Control in Manufacturing*). Another publication of the research described in this report can be found in the September 1996 issue of Journal of Materials (JOM). This paper was also published as a hypertext HTML document on the JOM web site:

http://www.tms.org/pubs.journals/JOM/9609/Stubbs-9609.html.

2. TECHNICAL WORK

2.1 Task I - Substrate Selection

A substrate that is stable at high temperatures and compatible with the piezoelectric film will be identified. It should have thermal, electrical, and mechanical properties that provide an optimum match with the high temperature piezoelectric film, and be capable of surviving the HIP cycle without contaminating the work piece.

Several substrates had been coated with aluminum nitride (AlN) film in brief trials prior to this project. Silicon carbide (SiC), tungsten carbide (WC), graphite, and a mixture of alumina and titanium carbide (Al/TiC) had shown some promise of being useful substrate materials. A search was conducted to identify suitable substrates by identifying key mechanical and thermal properties of materials able to withstand temperatures above 900 °C (1652 °F). After examining the pertinent properties, three substrates were selected for initial tests: graphite, tungsten carbide (WC), and silicon carbide (SiC). The table below lists some of their mechanical and electrical properties.

Table I - Properties of Candidate Substrate Materials

Substrate	CTE, °C (x 10^{-6})	Max. Use Temp.	Density (g/cc)	Resistivity	Modulus (GPa)	Acoustic Velocity (m/s)	Strength @ 1100°C (MPa)
WC	5-11	>900°C	11-15	Low	300-600	6,660	690
Graphite	1-2	*	2.25	Low	~ 8	4210	Low
SiC	5	1600 °C	3.21	Semi-cond.	340-400	2780	100-200

* Depends on the amount of oxygen in the environment.

Early in the project several attempts were made to deposit AlN onto SiC substrates without success. It was not understood why the attempts were unsuccessful, however, a possibility was that the silicon carbide (SiC) contained more residual carbon than was thought, thus preventing the AlN film from adhering well. A decision was made to choose tungsten carbide as the primary substrate for the sensor because of successful deposition of thick AlN films on three WC substrates. This decision meant the sensor was limited to a maximum temperature of approximately 1000 °C (1832 °F) when using WC as a substrate rather than temperatures as high as 1500 °C (2732 °F) that could be obtained using SiC.

Three grades of WC (containing 3 percent, 6 percent, or 12 percent cobalt) were chosen to have AlN deposited on them to test for differences in film adherence. All three grades readily accepted the AlN film with no apparent differences in adherence. The successful deposition of AlN on the three grades of WC prompted a search for alternate WC materials as candidate

substrates. Tungsten carbide, when alloyed with cobalt, oxidizes in air at temperatures exceeding 600 °C. Although this didn't present a problem in the argon atmosphere in HIP vessels, we preferred to design the sensor to withstand 1000 °C in air if possible. Other WC grades (such as those containing nickel) were identified that have improved oxidation resistance but lack of time prevented actual testing of these grades as substrate candidates.

The first tungsten carbide (WC) substrate cost over $650 to procure and machine into the shape required for the sensor. While investigating sources of different grades of WC a company was located (Vistametals, Inc. in Pennsylvania) that could produce a near-net shape substrate for approximately $25 using a pressed-powder and sintering technique. WC substrates were obtained from Vistametals and tested for compatibility with the AlN film deposition process. The material readily accepted the AlN film with good adherence. The final grinding of the near-net shape substrate to drawing specifications was done by Aggressive Grinding, Inc. (in Pennsylvania) for approximately $30.

The substrate design effort and materials selected in Task I proved very successful. Low cost (approximately $55 total) WC substrates were obtained that met the dimensional requirements of the sensor (Figure 1). The AlN film adhered well to the WC substrates and did not peel off at temperatures up to 1100 °C (2012 °F). The substrate did not deteriorate in any way when exposed to 1000 °C in an argon environment for over 30 minutes at pressures exceeding 155 MPa (22,500 psi). The electrical conductivity of WC proved to be sufficient to allow it to serve as one of the two electrodes for the AlN film. Figure 2 shows a photograph of several WC substrates with AlN deposited on the upward face.

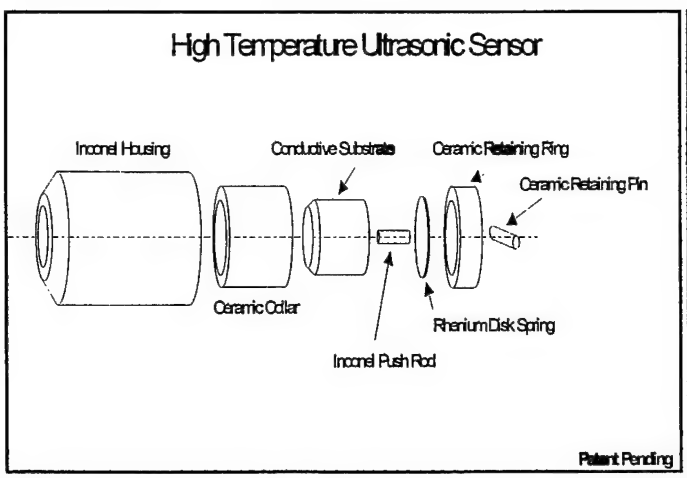

Figure 1 - The components of the sensor are shown. The aluminum nitride film is deposited on the forward face of the substrate.

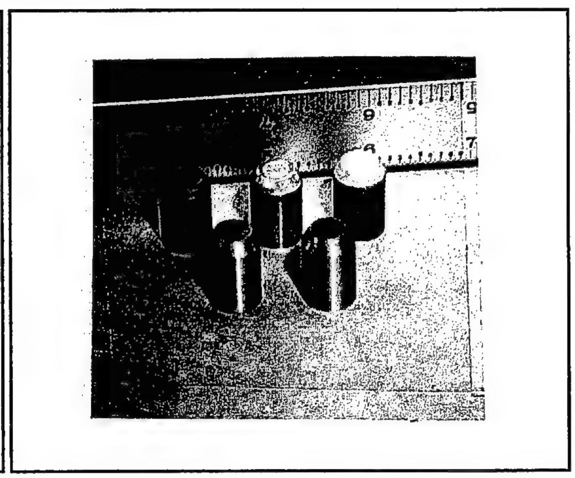

Figure 2 - Five tungsten carbide substrates are shown. The three substrates in the back have AlN deposited on their top faces.

2.2 Task II - Electrical Connections

Means for connecting the piezoelectric film with the electrical excitation source must be found such that the high temperature integrity of the film and connections are maintained.

The electrical connections from the ultrasonic pulser-receiver to the AlN film had to satisfy several requirements including withstanding temperatures above 1000 °C (1832 °F), maintaining satisfactory electrical impedance, tolerating strains from normal handling, allowing easy removal of the sensor's components, and (for some tasks) sustaining contact between the sensor and work piece. For safety reasons the sensor housing was the ground electrode for the pulser-receiver. The design of the electrical contacts to both faces of the AlN film were also considered as part of this task.

One of the first electrical connection decisions made was to use standard, off-the-shelf, thermocouple wire as the signal lines to and from the sensor. Sheathed thermocouple wire was obtained for electrical testing. The 1.6 mm (0.062 inch) diameter wire was Type K, sheathed in Inconel® 600 cladding, and insulated with magnesium oxide. The manufacturer's specifications indicated a maximum temperature range of 1148 °C (2100 °F) for this configuration. Tests of the propagation of ultrasonic-frequency, electrical signals in the thermocouple wire showed losses of less than 17 dB per meter which was considered acceptable.

The Inconel® sheath served as the ground electrode by being clamped to the sensor housing. The thermocouple wires were used as the signal carrying electrode and were welded to an Inconel® "push rod" that fit into a hole in the back of the substrate. Electrical continuity to the back face of the AlN film was achieved through the contacts between the push rod and the conductive substrate.

The most complicated part of the electrical connection task involved applying a conductive film to the outer face of the AlN film. Initially, sputtered platinum was tried but difficulties were encountered in producing a thick film, especially on the bevelled faces of the substrate. A second method of plating the outer face of the AlN film was examined and found to be successful. Platinum ink, which can be painted onto metallic and glass surfaces and then fired to a hard, conductive coating, was applied to the AlN film. This method was used on one of the prototype sensors.

Ultimately, two modifications to the sensor design were created to improve the electrical connection between the sensor housing and the platinum coated outer face of the AlN film. A cap was fixed to the front of the sensor which made a direct physical contact with the platinum film. The cap was screwed onto the housing so the pressure the cap applied to the film could be adjusted. This modification worked well to ensure a strong electrical connection to the front of the AlN film. In addition, a thin foil of platinum was inserted between the cap and substrate to aid in maintaining a good electrical connection.

2.3 Task III - Ultrasonic Coupling

Research shall be conducted to provide a method of coupling the ultrasonic energy to the HIP work piece at temperatures and pressures typically encountered in HIP vessels.

Two parallel approaches were followed to find a means of coupling the ultrasonic energy from the sensor to the work piece inside the HIP vessel: 1) identification of potential high temperature couplants, and 2) examination of the potential of using the high pressure argon inside the HIP vessel as the couplant. The intent was to find a method that would allow efficient coupling of the ultrasonic energy to the work piece but not contaminate the HIP vessel or sensor.

Initially, we felt that the propagation through the high density argon gas approach (referred to as the "argon gas method" hereafter in this report) had a lower chance of success than using appropriate melting point glasses or metals. However, the research ultimately showed that the high pressure argon gas provided very efficient coupling.

A search for potential metals and glasses for coupling ultrasonic energy in the temperature range of 600 °C to 1000 °C produced the candidates listed in Table II. The four elemental metals and three glasses similar to those listed in Table II were obtained. One of the glasses (Borosilicate) was tested as a couplant in a open-air furnace. The glass softened as expected and appeared to form a suitable coupling layer between the AlN film and a metal target. However, some of the glass deposited onto the furnace's thermocouple wires through a gas transport mechanism. Consultation with operators of HIP vessels raised the concern that the use of metals and/or glasses inside the HIP vessels could produce contamination and possibly damage to the vessel. The use of high melting point couplants was put on hold until results from the argon gas method could be examined.

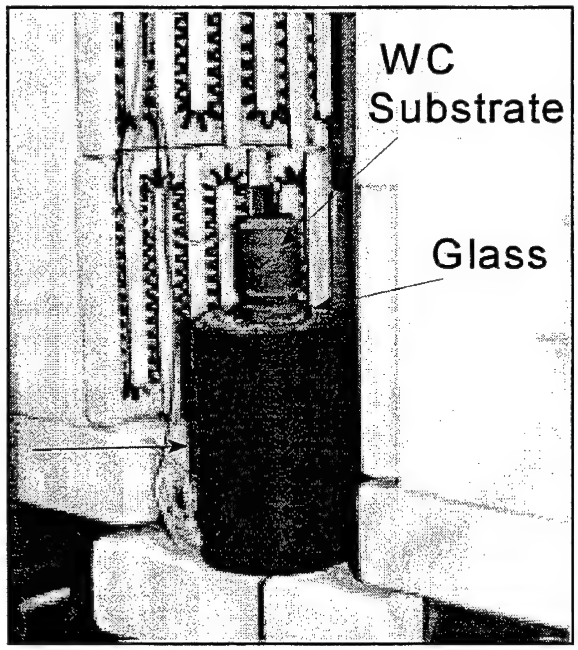

Figure 3 - Borosilicate glass was tested as a high temperature couplant. Although deficiencies in the AlN film prevented complete testing of the glass, the results indicated that the glass could be used as a couplant.

Table II - Properties of Materials to be Investigated for Use as High-Temperature Ultrasonic Couplants

Material	Melting Pt. (°C)	Solidus Pt. (°C)	Boiling Pt. (°C)	Form	Cost for 25 x 25 mm	Source
Aluminum	660		2467	foil, 0.03 mm	$21	Aesar
Cerium	799		3426	foil, 0.1 mm	$89	Aesar
Lanthanum	921		3457	foil, 0.1 mm	$74	Aesar
Silver	962		2212	foil, 0.03 mm	$16	Aesar
92.5 Al, 7.5 Si	610	577		film		Indium Corp.
72 Ag, 28 Cu	780	780		film		Indium Corp.
82 Au, 18 Ni	950	950		film		Indium Corp.
	Softening Pt. (°C)	**Working Pt. (°C)**				
Borosilicate 7052	712	1128		powder	N/A	Corning
Soda Lime 0080	696	1005		powder	N/A	Corning
Duran 8330	815	1270		powder	N/A	Corning
Aluminosilicate 1723	915	1202		solid block	N/A	Corning

Fortunately, experiments showed that the pressurized argon gas inside the vessel was a very efficient coupling medium for ultrasound in the 15 to 50 MHZ range. Measurements were taken of the reflected amplitude and the velocity of ultrasound through pressurized argon gas at 77 °C (170 °F) using transducers having center frequencies of 15, 20, and 25 MHZ. The ultrasonic energy was directed towards a 12.7 mm thick, stainless steel mirror that was positioned at various distances from the transducer. Amplitude and time-of-flight measurements were acquired as the gas pressure was increased from atmospheric pressure to approximately 200 MPa (~30,000 psi). Data were acquired during pressurization and during venting of the HIP vessel. For each measurement of the ultrasonic parameters the pressure and temperature of the gas in the HIP vessel were also recorded. The entire pressurization cycle took approximately 5 hours.

Reflections from the mirror were observable on an oscilloscope when the argon gas pressure was as low as 12 MPa (1800 psi) and at higher pressures multiple reflections from the back surface of the mirror were observable. A reflection from a 1.5 mm diameter hole, drilled midway deep and parallel to the plane of the mirror, was observable in many of the signals

(Figure 4). It was found that ultrasonic pulses could be reflected off the stainless steel mirror up to 140 mm away, with signal-to-noise ratios exceeding 3:1, when the pressure inside the HIP vessel was greater than 35 MPa (5000 psi).

Initially, the time-of-flights, and corresponding velocity calculations, were compared with ideal gas law predictions using the following equation:

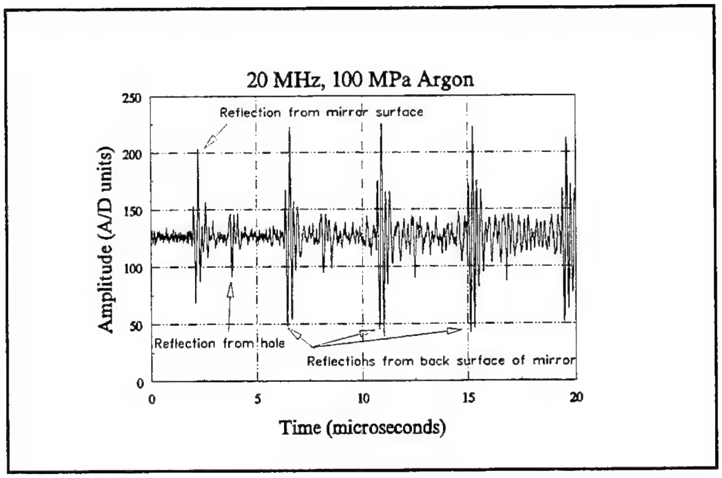

Figure 4 - Reflections of 20 MHZ ultrasound from a 12.7 mm thick stainless steel mirror, 82 mm from the transducer. The ultrasonic signal was acquired after a 230.0 microsecond delay. Echoes from the back surface of the mirror as well as from a 1.5 mm diameter holed drilled through the mirror are clearly visible in the signal. The temperature inside the HIP vessel was 77 °C (170 °F).

$$V = \sqrt{\frac{P * \gamma}{\rho}} \tag{1}$$

where V = velocity of sound
P = gas pressure
γ = specific heat constant
ρ = density of gas

The density of argon gas as a function of pressure was taken from various tables and graphs. Little data were readily available for the specific heat constant of argon as a function of pressure. The specific heat constant is the ratio of the specific heat of argon at constant pressure to the specific heat of argon at constant volume:

$$\gamma = \frac{C_p}{C_v} \tag{2}$$

Again, initially an estimate of 1.67 was used for γ; this value was known to be correct for argon gas a room temperature and atmospheric pressure. However, it was also known that C_p and C_v

change as a function of pressure and temperature so it was likely that the value of 1.67 is not correct for higher pressure argon gas. In addition, the argon gas temperature increased to approximately 77 °C (170 °F), as the HIP vessel was pressurized. Since C_p and C_v have a temperature dependence γ probably changed as the temperature increased.

Figure 5 shows time-of-flight and velocity data from the ultrasound reflected from the stainless steel mirror at a distance of 82 mm. The 20 MHZ transducer was used to acquire the data for this graph. The solid symbols represent the measured values and the hollow symbols represent the predicted values based on Equations 1 and 2 using $\gamma = 1.67$. The graph shows that the calculated velocity values, as a function of gas pressure, always exceeded the predicted velocity values. This was consistent for all of the experiments conducted; the ultrasonic velocity data (for different frequencies and different transducer-to-mirror distances) followed the trend but exceeded the values of the predicted data.

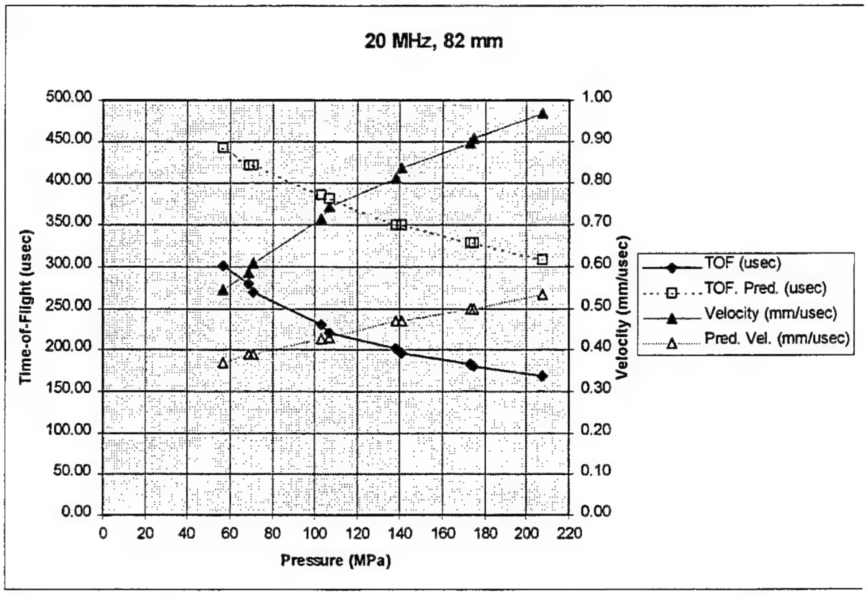

Figure 5 - Predicted and measured values for the time-of-flight and velocity of ultrasound through 164 mm (82 mm distance between transducer and mirror) of argon gas, as a function of gas pressure.

It was important to understand the difference between the predicted and measured time-of-flight (or velocity) data. A thorough understanding of the velocity of ultrasound through argon gas as a function of pressure and temperature would allow accurate measurement of the distance from the transducer to the parts being HIPed without relying on a calibration procedure to correct for the changes due to pressure and temperature. Additional searching of the literature revealed several papers that contained experimentally-obtained data on the velocity of sound in high pressure argon [2-]. In addition, a book by Van Itterbeek, titled "Physics of High Pressures and the Condensed Phase", contained detailed derivations of various thermodynamic models used to predict the velocity of sound in argon and other gases. One model, called the "virial coefficient" model, made use of empirically-derived coefficients to calculate the velocity of sound in argon at pressures and temperatures near those present in a HIP vessel.

The virial coefficient model was programmed into Excel to allow curves of velocity versus pressure to be made. Figure 6 shows the velocity of sound predictions using the virial

coefficient model for several temperatures from room temperature to over 400 °F (205 °C). The single data points (not connected by a line) represent experimental data acquired using 20 MHZ ultrasound inside a HIP vessel at a temperature of approximately 77 °C (170 °F). The data follow the general prediction of the virial coefficient model for temperatures under 200 °F up to a pressure of 100 MPa. At higher pressures the data tend towards the virial coefficient model for a temperature between 200 and 400 °F. This correlation between experimental data and the virial coefficient predictions was strong enough to use the virial coefficient model for subsequent HIP runs.

Of interest is the trend of the velocity versus pressure curves to have a smaller slope for higher temperatures. The large decrease in slope between 188 °F (87 °C) and 403 °F (206 °C) in Figure 6 suggests that the curve at HIP temperatures (typically above 600 °C (1112 °F)) might have zero slope or even be negative. In fact, HIP vessel tests at 150 MPa in which the argon gas was at temperatures of 150 °C , 287 °C , and 576 °C yielded velocity of sound measurements of 0.77 mm/µs, 0.76 mm/µs, and 0.75 mm/µs respectively. Referring to Figure 6 shows that these velocity values fall on the 403 °F (206 °C) curve. Obviously, additional research on the virial coefficient model needs to be done for successful use in production HIP vessel runs.

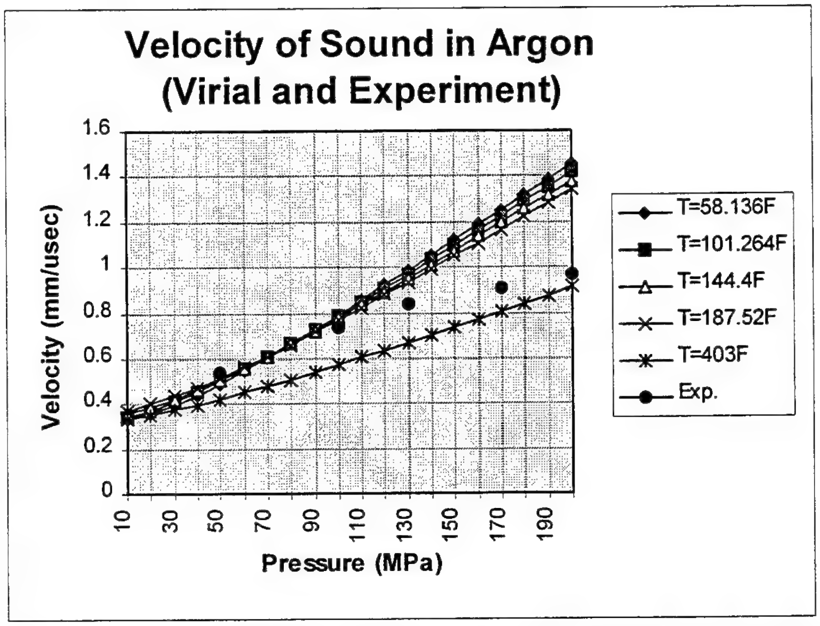

Figure 6 - The predicted velocity of sound in pressurized argon gas is shown as a function of gas pressure for several temperatures. The single data points not connected by a line represent actual measurements.

For more information on the virial coefficient model the reader is referred to Physics of High Pressures and the Condensed Phase, A. Van Itterbeek, Chpt. 7, section 2.

2.4 Task IV - Transducer Housing

A transducer housing that provides mechanical stability for the substrate and film, electrical connections, and fixturing to the HIP vessel should be created.

The initial design goal for the transducer housing was to produce a protective, rugged enclosure for the aluminum nitride film, substrate, and electrical connections. Specifically, the design addressed:

a) keeping the housing and substrate as separate components so that substrates could be removed and inserted without damaging the housing,

b) making the housing the ground electrical connection for the AlN film. The electrical conduction was made between the beveled side of the substrate and the angled rim of the housing,

c) holding the substrate in place using an insulating ceramic sleeve and a spring - loaded assembly that matched the thermal coefficient of the housing,

d) consideration of the thermal expansion, high temperature strength, oxidation, and ease-of-manufacturing properties of all housing components.

A nickel-based superalloy, Inconel®625, was selected as the housing material because of its high temperature stability and ease of machining characteristics. Careful design resulted in relatively straight-forward machining of the housing at a reasonable cost and simple disassembly to replace any components. The design also allowed off-the-shelf components to be used for electrical connections and insulation purposes. Figure 7 shows the housing design used in the prototype sensor. The ceramic collar electrically insulates the substrate from the housing. The rhenium spring and Inconel®625 push rod kept the substrate pushed against the front of the housing. Since the thermal expansion of the housing was matched by the expansion of the push rod, the elongaton changes due to the thermal expansion of the housing and push rod were

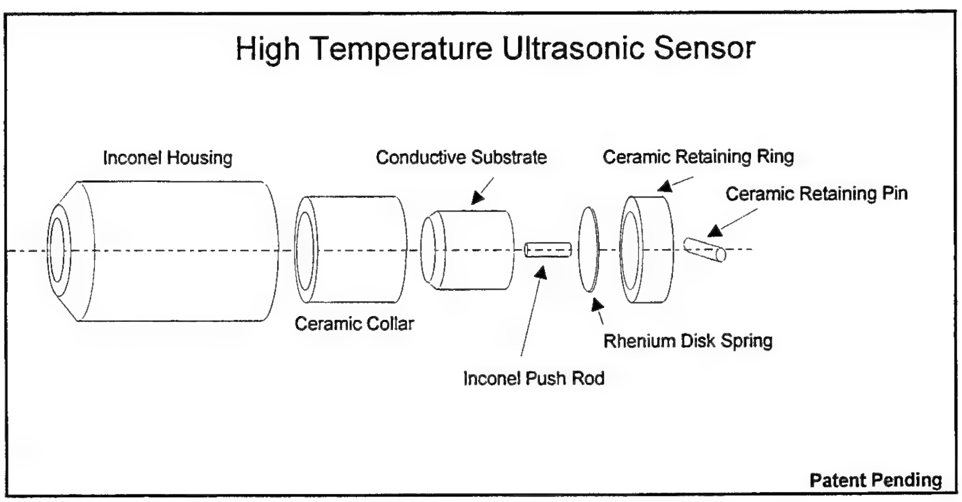

Figure 7 - The schematic drawing shows the components of the sensor. The AlN film was deposited on the left-hand face of the substrate.

constant regardless of temperature. Some changes in the fixturing forces on the substrate were created due to the expansion of the substrate and variation of the modulus of the rhenium spring as a function of temperature. The ceramic retaining ring and retaining pin held the spring assembly in place within the sensor.

Inconel® 625 was selected because of its high temperature properties, good oxidation resistance, and good resistance to corrosive environments. For comparison, Inconel® 625 is equal or superior to Inconel® 718, Incoloy® 800 and 900 series, and the Monel® alloys in its high temperature, oxidation, and corrosive environment properties. Based on published properties of Inconel® 625 the housing should be useful at temperatures exceeding 1200 °C (2192 °F).

The rhenium material to fabricate the push rod spring was cut by wire electro-discharge machining to fit the housing. The rhenium was found to have the flexibility necessary to act as a high temperature spring in the sensor. The push rod was cut from stock Inconel® 625 rod.

The ceramic collar and retaining ring were cut from stock alumina tubing. The ceramic retaining ring had a seating ledge for the rhenium disk to center and hold the disk in the sensor.

A strain-relief mechanism was designed to protect the electrical connections coming into the sensor from the pulser-receiver. The design goals were:

a) To provide a rugged strain-relief for the electrical connections to the sensor,
b) To allow convenient insertion of new electrical connection wire, and
c) To provide an area on the sensor for attachment to other fixturing devices.

Figure 8 shows the strain-relief design. The ring around the sensor has a drilled-through hole (without threads) that the bolt fits through. The bolt screws into a clamping plate that contains a groove to position the electrical wire. The screw and clamp plate can be disposed of if the high temperature environment causes them to be damaged during removal while the ring around the sensor remains undamaged. In the second generation prototype sensor the ring was machined as part of the sensor instead of fitting it to the sensor as a separate assembly step.

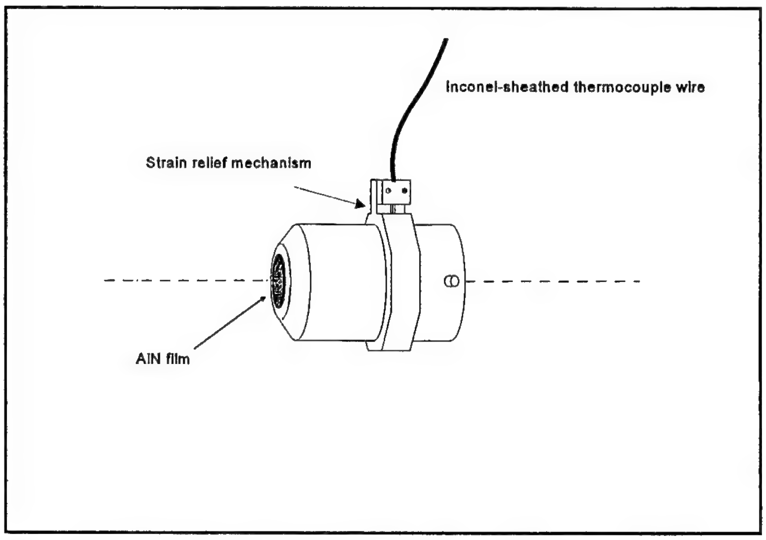

Figure 8 - The strain relief mechanism for the thermocouple wire that served as the signal carrier is shown.

Due to problems with electrical connections between the inside of the bevelled region of the housing and the AlN film on the bevelled region of the substrate (see Task II for a more detailed description) the sensor housing was modified during the design of the second generation prototype. A cap was fitted to the housing to make a firmer connection between the housing and the forward face of the AlN film (see Figure 9). While this modification was not aesthetically pleasing, it did serve its purpose.

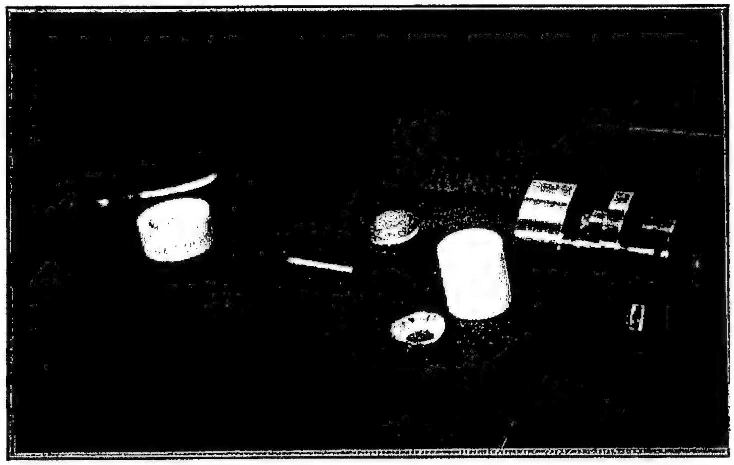

Figure 9 - The housing and sensor components are shown in this photograph. The housing has been fitted with a screw-on cap to improve the electrical connection to the front of the AlN film.

2.5 Task V - Prototype Transducer

A prototype transducer shall be built using the knowledge gained from the research tasks.

Two prototype transducers were designed, constructed, and used for various tests before the final prototype transducer was designed and used in HIP vessel tests. Each prototype was designed to incorporate improvements resulting from the work in each of the four research tasks described previously in this report. Figure 10 shows a drawing of the first prototype sensor used to test electrical performance of the insulating ceramics, thermocouple wire, and substrate. Figure 11 shows a photograph of the second prototype sensor used to test all components of the sensor. This design was used extensively to optimize the platinum coating on the outer face of the AlN film. The final prototype sensor, used in all elevated temperature tests in and out of the HIP vessels, is shown in Figure 12.

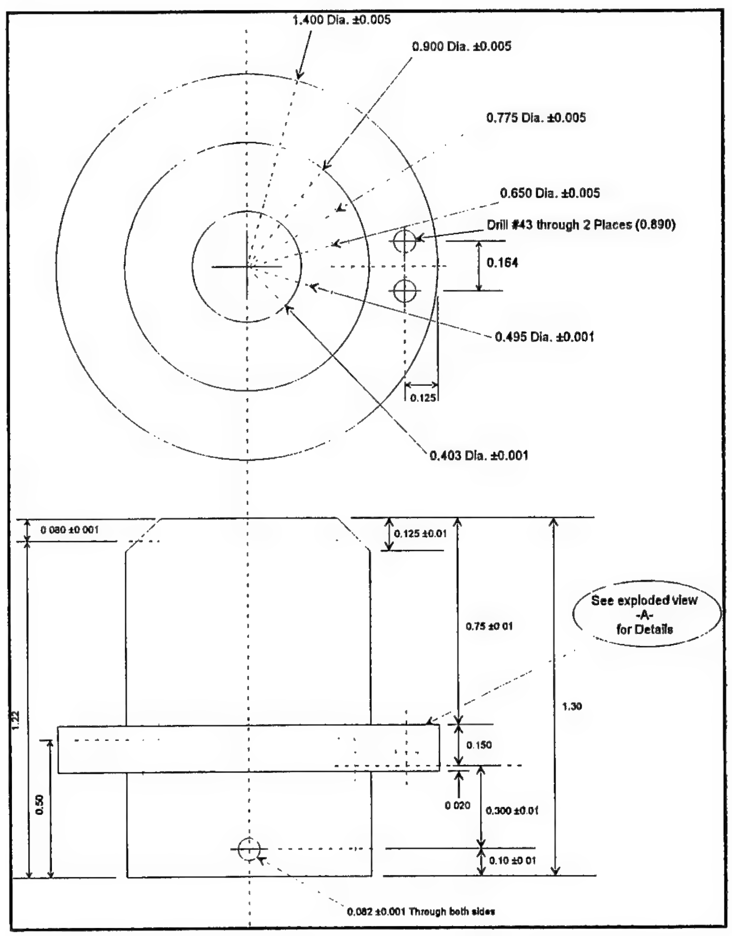

Figure 10 - Dimensional drawing of the first prototype sensor housing.

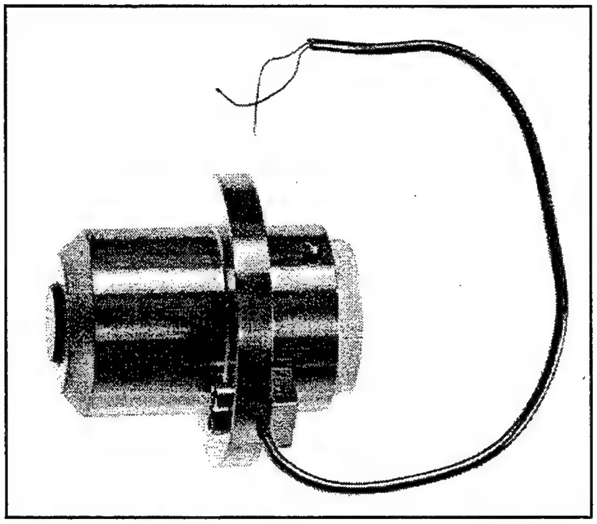

Figure 11 - Second prototype sensor design.

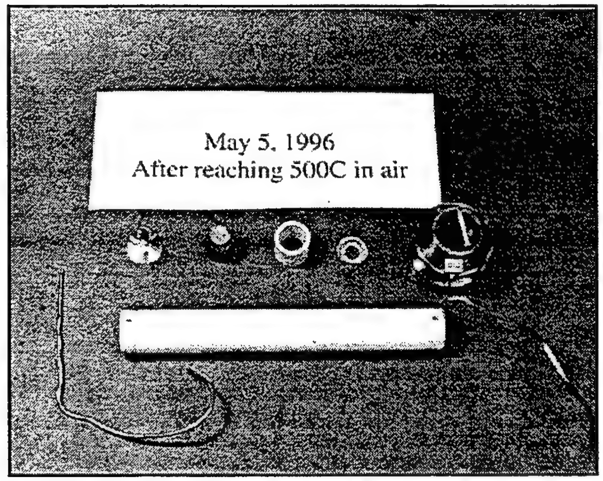

Figure 12 - The final sensor design is shown in this photograph taken after the sensor was tested at 500 °C (932 °F) in air.

2.6 Task VI - HIP Vessel Tests to Demonstrate Work Piece Deformation Monitoring

The prototype transducer shall be used in a HIP vessel, under typical HIP cycle conditions to monitor the deformation process of the work piece. The goal will be to detect changes in the size of the HIP Can on the order of 0.2 mm. This can be accomplished by having the transducer in contact with the HIP work piece.

and

2.7 Task VII - Demonstration of the Deformation Measurement Using Echo Ranging

The prototype transducer shall be used in a HIP vessel to detect deformation of the HIP work piece using an echo-ranging method.

Task VI was designed to demonstrate that the sensor could monitor the deformation of a work piece in the HIP vessel with a deformation resolution of 0.2 mm or better. The goal was to demonstrate the sensor performance under typical HIP conditions: temperatures around 900 °C (1652 °F) and pressures at 100 MPa (14,500 psi). If the argon gas propagation mode was used, deformation measurements were to be demonstrated by measuring the time-of-flight of the ultrasound through the gas and multiplying by the estimated velocity of sound for the given temperature and pressure. If the contact mode of ultrasonic coupling was used, deformation measurements could have been monitored by detecting the reflection of ultrasound from the back side of the target. However, it was anticipated that the back surface echo would be considerably

weaker than the front surface echo and may have been impossible to detect.

The results from Task III (Ultrasonic Coupling) resulted in the use of the argon gas method for ultrasonic coupling in the HIP vessel tests. Successful completion of Task VI meant demonstrating the capability of the sensor (and the entire ultrasonic system) to measure the ultrasonic echoes with enough resolution to monitor deformations of 0.2 mm or better under typical HIP run conditions. Two tests of the sensor in the HIP vessel were conducted to fulfill the requirements of Task VI (and Task VII). While neither test alone completely demonstrated reaching the Task VI goals, taken together, the two tests demonstrated the capability of the sensor to monitor work piece deformation on the order of 0.001 mm during typical HIP runs.

As an example of the requirements for monitoring deformation with a resolution of 0.2 mm, consider the HIP runs where the ultrasonic reflection from a target 33 mm away was acquired. To achieve a deformation resolution of 0.2 mm, the time-of-flight of the echo must be measured with a time resolution of 0.4 microseconds according to Equation (3):

$$\Delta T = \frac{2 * \Delta X}{V} \qquad \textbf{(3)}$$

Where ΔX = 0.2 mm and
V = 1 mm / μs (a relatively high value for the velocity of sound in pressurized argon gas - see Figure 5)

Ultrasonic reflection data acquired during the HIP runs actually produced deformation resolutions several orders of magnitude better than this. The system analog-to-digital convertor sampled the ultrasonic signals at 500 MHz, or at 0.002 micro-second intervals. Inserting 0.002 for ΔT in Equation (3) and using a value of 0.75 mm/μs for the velocity of sound (a typical value at elevated temperatures and 150 MPa in the HIP vessel) yields a theoretical deformation measurement resolution (ΔX) of 0.0008 mm for the tests run in the HIP vessel.

The original intent of Task VII, as stated in the SOW, was to make use of the gas propagation mode, even if it was shown to be inferior to the contact coupling mode, to demonstrate some capability of the method. When the SOW was written it was thought that the gas propagation mode could be made to work, but might be possible only for very short propagation distances. As explained earlier in this report, propagation of ultrasound was found to be possible over distances exceeding 100 mm in the pressurized argon gas.

3. HIP TEST RESULTS

Two different ultrasonic signals from the sensor were monitored and acquired during HIP vessel testing. One signal represented the ultrasonic reflection from the back of the substrate. The other signal represented the reflection from a nickel-based superalloy target 33 mm in front of the sensor. The ultrasonic energy reached the target by propagating through the argon gas. During the first test the signal from the back of the substrate was monitored while the pressure and temperature were increased. In the second test both the substrate reflection and the reflection from the target 33 mm away from the sensor were monitored.

During the first test the pressure in the HIP vessel was ramped to 150 MPa (~22,000 psi) while the temperature was held at 65°C (150°F). After the pressure reached 100 MPa the temperature was increased to 1000°C (1832 °F) at a rate of 10°C (18 °F) per minute. During the temperature ramp the pressure increased to over 155 MPa (22,500 psi). Figure 13 shows the amplitude of the ultrasonic reflections from the back of the substrate as a function of temperature. The amplitude varied somewhat as the temperature increased but stayed at approximately 60% full scale (150 digitizer counts) up to 850°C (1562°F). The last data were

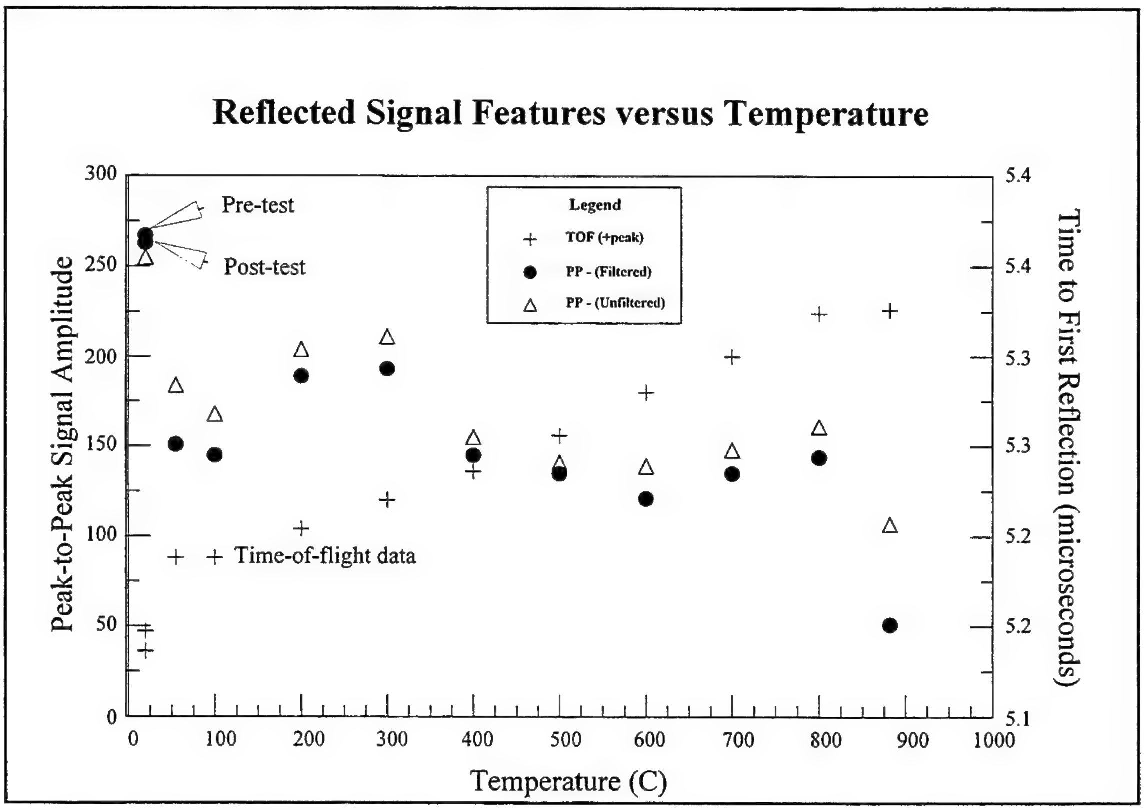

Figure 13 - Amplitude and time-of-flight data from the reflection from the back of the substrate are shown as a function of temperature inside the HIP vessel. The pressure was 150 MPa or greater for all temperatures above room temperature.

recorded at 882°C (1617°F) and the signal was detectable up to 940°C (1724°F). The temperature in the HIP vessel was increased to 1000°C (1832°F) before cooling down to room temperature. The sensor after removal from the HIP vessel is shown in Figure 14. The sensor and its components received a coating from the oxidation of a structure used to support the sensor, but were otherwise unaffected by the high temperature/high pressure exposure. The data recorded at room temperature were obtained with the sensor outside the HIP vessel and connected directly to the pulser-receiver. This was true for both data points acquired at room temperature - before and after the HIP run. All other data were acquired with the sensor inside the HIP vessel. The lower signal amplitudes for all data taken above room temperature were due to signal losses resulting from connecting the sensor to the pulser-receiver using thermocouple wires passing through the HIP vessel wall.

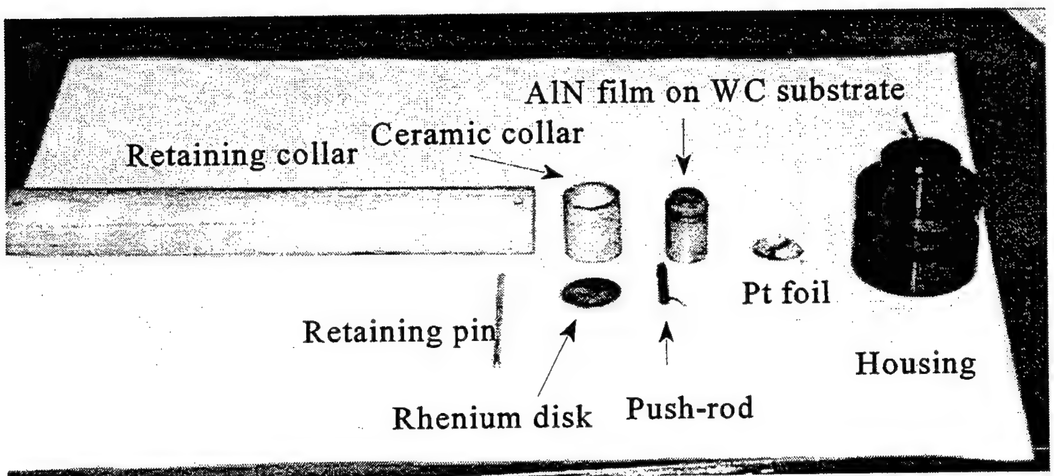

Figure 14 - This photograph was taken of the sensor's components immediately after removal from the HIP vessel. The sensor was heated to 1000°C and subjected to pressures exceeding 155 MPa during the HIP run. No structural damage was found on any of the sensor's components.

The second HIP run was conducted with the sensor positioned so that the ultrasound emitted from the front face of the AlN propagated through 33 mm (1.3 inches) of argon gas to a nickel-based superalloy target. The test started by ramping the pressure to 100 MPa (14,600 psi) at which point a reflection from the target was recorded. The pressure was increased to 155 MPa (22,500 psi) before beginning the temperature ramp. The temperature in the HIP vessel was increased to 1100°C (2012°F) at a rate of 20°C per minute while holding the pressure constant at ~150 MPa. Ultrasonic data were recorded at approximately 100°C intervals. Figures 15a and 145 show the ultrasonic signal propagating through the argon gas to the target and back at 150°C (302°F) and 576°C (1060°F) respectively. At 576°C the signal amplitude was small although still useful for detecting the presence of the target. The reflection from the target was

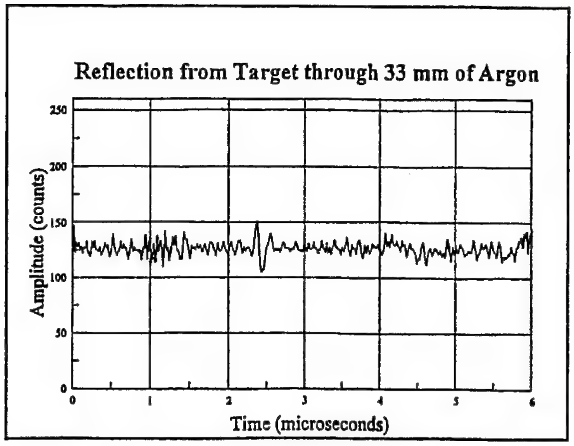

Figure 15a - The reflection from a target 33 mm away from the transducer is shown at 2.5 microseconds. The sensor was in a HIP vessel at 150°C and 150 MPa.

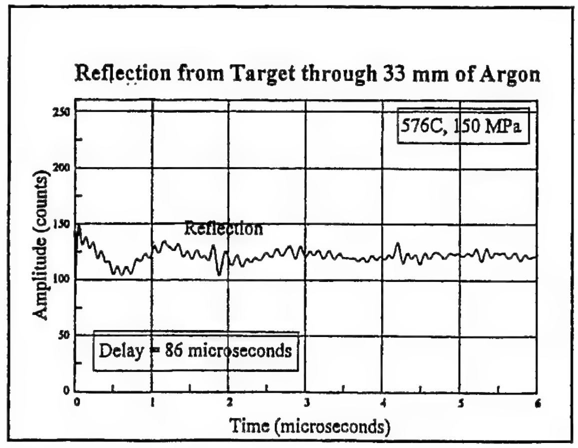

Figure 15b - The reflection from the target is shown at approximately 2 microseconds. The HIP vessel temperature was 576°C and the pressure was 150 MPa.

detectable up to 676°C, after which it was overwhelmed by electrical noise. The sensor continued to emit ultrasound up to a temperature of 940°C (1724°F) at which point electrical noise overwhelmed the substrate reflection signal making identification of the ultrasonic reflections impossible. Figure 16 shows a reflection from the back of the substrate at 882 °C (1617 °F) and a pressure of 75 MPa (10,900 psi). The HIP run was continued to 1100°C and then cooled to room temperature. After removing the sensor from the vessel it was determined that the electrical noise arose due to deterioration of the electrical connections to the AlN film. Subsequent testing of the AlN film and substrate, and visual inspection of the housing (see Figure 17) and internal components revealed no other damage from the 1100°C temperature.

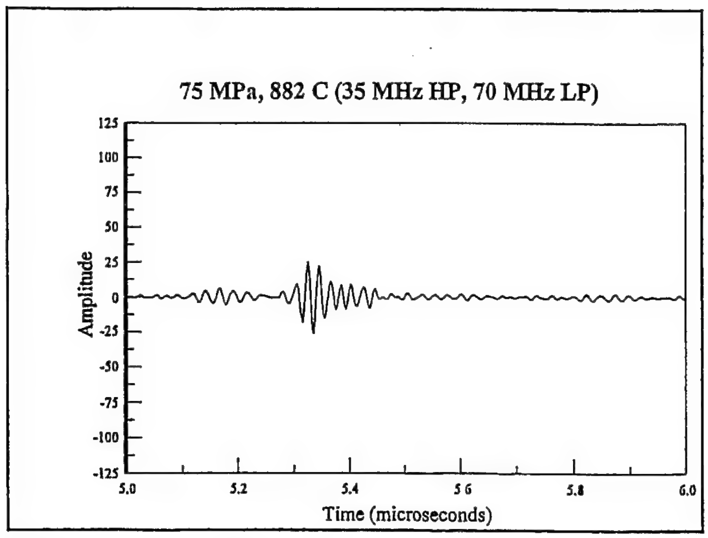

Figure 16 - A reflection of ultrasound from the back side of the substrate was recorded at a temperature of 882 °C and an argon gas pressure of 75 MPa. This signal was filtered after acquisition using 35 MHZ high pass and 70 MHZ low pass filters.

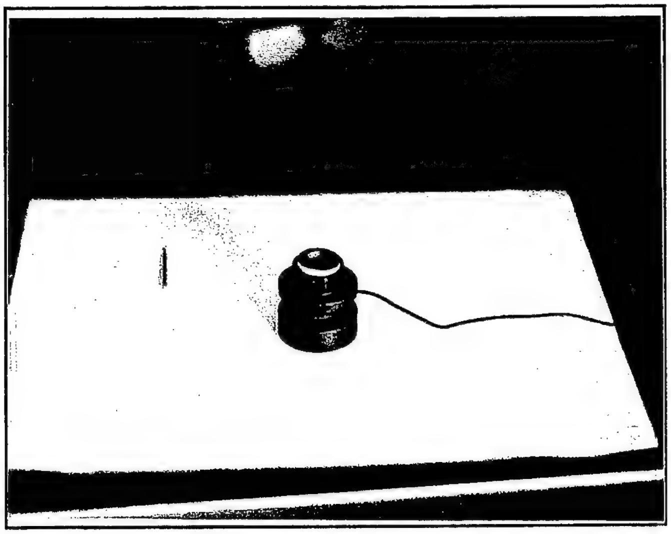

Figure 17 - This photograph shows the sensor after removal from a HIP run in which the temperature reached 1100 °C and the pressure exceeded 150 MPa. No structural damage to the sensor housing or internal components was observed.

4. CONCLUSION

The goals of all seven tasks in the state of work were accomplished. An ultrasonic transducer was developed that is suitable for use as a displacement sensor in the high temperature and high pressure environment typically found in a HIP vessel. The sensor was tested to 1100°C (2012°F) and 150 MPa (22,000 psi) without sustaining damage and emitted useable ultrasound at temperatures above 900°C (1652°F) before electrical connection problems occurred.

There were several problems and/or areas needing improvement encountered during the project that should be mentioned in order to alleviate problems if future work is done with this type of sensor. In order of importance they were:

1) Delivery of the AlN films. UDRI received only two AlN films deposited on machined WC substrates in time to be useful for HIP testing. Six AlN films on machined substrates were received one day before the end of the technical portion of the project. The shortage of useable AlN films deposited on substrates limited the number of HIP runs that were done.
   There is currently only one source of AlN of the type necessary for high temperature ultrasonic sensors. UDRI is investigating acquiring the technical expertise, equipment, and any rights in technology necessary to make the films.

2) Coating the outer face of the AlN film with a conductive, high temperature, metallic coating. The use of platinum paste was successful, but more work needs to be done to create a rugged, thick, platinum film in order to make a sensor suitable for use at HIP facilities by HIP company personnel.

3) Electrical noise occurred during both HIP runs when the temperature approached 700 °C (1292 °F). It was felt the mostly likely candidate for causing this problem was the degradation of the Inconel®-sheathed thermocouple wire, although several other components could also cause electrical noise problems. Additional work should be done to try to reduce or eliminate the electrical noise. Also, the use of bandpass filters should be used during future tests to reduce the effects of the noise.

4) A better understanding of the virial coefficient model for propagation of sound through high density argon gas is needed to accurately measure deformation during HIP runs. The model was relatively successful in predicting the trend of the velocity of sound in argon gas at different pressures and temperatures. A thorough understanding of the model, and extension to the temperatures encountered in HIP runs, is necessary before the technique could be used in day-to-day HIP processing.

Due to the nature of the piezoelectric properties of the AlN film it is expected that the film can be used at temperatures exceeding 1200°C (2192°F). The current design for the entire sensor also should allow its use at temperatures approaching 1200°C.

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APPENDIX A

"An Ultrasonic Sensor for High Temperature Materials"

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AN ULTRASONIC SENSOR FOR HIGH TEMPERATURE MATERIALS

PROCESSING

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ABSTRACT

A sensor has been developed and tested that is capable of emitting and receiving ultrasonic energy at temperatures exceeding 900°C and pressures above 150 MPa. The sensor works with standard ultrasonic pulser-receivers and has demonstrated the capability of measuring workpiece deformation during hot isostatic pressing (HIP). Details of the sensor design, performance, and coupling of the ultrasound to the workpiece are described. Ultrasonic data acquired by the sensor, *in situ*, during HIP runs and at elevated temperatures in air are presented.

INTRODUCTION

High temperature structural components, such as structural castings for jet engines, nickel-base superalloy turbine blades, and ceramic and metal matrix composites made from powders are often densified by hot isostatic pressing (HIP). For powder consolidification, the HIP process involves sealing the powder (and reinforcing fibers for composites) within an evacuated "Can" made of a material that softens at high temperatures. A hydrostatic pressure is then applied by pressurizing the inside of the HIP vessel with argon gas (up to 140 MPa). The hydrostatic pressure densifies the powder through the elimination of the porosity between the powder particles.

This paper presents details of the development and preliminary testing of an ultrasonic displacement sensor that can be used to monitor materials processing, *in situ*, at temperatures and pressures significantly higher than current ultrasonic displacement sensors. The target processing environment for this sensor is that typically found in a hot isostatic pressure (HIP) vessel, i.e., temperatures exceeding 1000°C and pressures above 138 MPa.

CURRENT ELEVATED TEMPERATURE DEFORMATION SENSORS

Dilatometers that can measure the *in situ* deformation of the HIP work piece during the process cycle have been developed for research applications (1,2,3) but these are limited to simple cylindrical shapes of small size. Dilatometers also are susceptible to malfunctions due to their mechanical nature. Because of the high temperatures and pressures, the only other instrumentation used in industrial HIP applications has been limited to thermocouples that monitor the external temperature of the HIP work piece. Since it is very difficult to reliably monitor the deformation during the HIP cycle with existing technology, it is typical practice to use the maximum rated pressure of the vessel and to hold at the processing temperature for an extended time to be completely confident that the work piece is fully dense. For some large geometries (such as those developed for NASP structural components) the HIP cycle can last over 24 hours when heat-up and cool-down times are included and cost up to $10,000 per run. In addition, if the HIP "Can" develops a pinhole leak or tear (e.g., due to deformation of the outer wall of the can over a sharp edge) the densification stops due to the equalization of pressure between the exterior and interior of the HIP Can. At this time it is not possible to determine if this has happened until the end of the HIP cycle. Since these leaks often develop during the initial pressurization, the entire run is wasted and must be repeated or scrapped.

A reliable sensor that could provide information on the actual consolidation of the material would save HIP time and reduce costs. Further, HIP runs could be terminated as soon as consolidation is complete reducing undesired effects caused by holding at elevated temperatures for extended periods of time (e.g. reactions between the fiber and matrix in metal-matrix composites). Recent progress has been made in adapting eddy current sensor technology to HIP vessel consolidation (1). However, this technology is limited in the range of displacement that can be measured, typically less than 20 mm, and the high cost of the probes.

Standard, room temperature, ultrasonic technology can measure displacements on the order of 0.02 mm over ranges exceeding 100 mm. Presently, commercially-

available ultrasonic transducers are limited to approximately 350°C for sustained, *in situ* use because of transducer materials. Extending ultrasonic displacement measurement to an elevated temperature environment presents many difficulties. The piezoelectric materials used in typical ultrasonic transducers become inefficient and may lose their piezoelectric properties if temperatures exceed the Curie point of the material. Typical efforts to adapt standard ultrasonic transducers to high temperatures use a "buffer rod" between the transducer and the hot material. For HIP vessel use a buffer rod is unacceptable due to the necessity of breaching the vessel wall.

Several methods of producing ultrasonic energy, other than using piezoelectric materials, have been developed. Electromagnetic acoustic transducers (EMATS) have been used successfully to detect defects and measure mechanical properties of metals at high temperatures (5). However, one drawback to using EMATS is the requirement that the target material be electrically conductive. Also, EMATS are generally very inefficient producers of ultrasonic energy so the resulting signal-to-noise ratios are often small. Laser technology has also been used to create and receive ultrasonic energy in a variety of materials (6). This technique shows considerable promise for producing ultrasonic energy in materials at elevated temperatures, but currently is very expensive and requires the use of moderate power lasers. In addition, the application of the laser energy often causes minor damage to the surface of the test object which is undesireable in many materials. Hence the laser ultrasonic and EMAT techniques, while useful for defect detection and characterization of mechanical properties, are not easily adapted for measuring the displacement of a workpiece in a HIP environment.

HIGH TEMPERATURE SENSOR DEVELOPMENT

As early as 1976, aluminum nitride was known to possess piezoelectric properties at high temperatures (7). In 1990 an AlN film was shown to produce ultrasonic energy at temperatures exceeding 1150°C for 24 hours with no degradation in the output of the ultrasonic energy (8). Recognizing the potential applications of a sensor based on AlN, the University of Dayton Research Institute (UDRI) and the U.S. Air Force began investigations into the requirements for a robust sensor suitable for industrial applications. These included the identification of a suitable substrate material and the development of both a high temperature sensor housing and electrical connections capable of withstanding the temperatures and pressures characterisitic of the HIP environment.

The requirements for a suitable high-temperature substrate included a thermal coefficient of expansion compatible with the AlN film, stability at high temperature, good adherence of the AlN film, and high electrical conductivity (so that the substrate serves as an electrode for the AlN film.) After investigating several materials, tungsten carbide was selected due to the good adherence of the AlN film and machining/manufacturing considerations. An Inconel alloy was selected as the housing material because of its high temperature stability and ease of machining characteristics. Achieving electrical connections to the AlN film that were stable at high temperatures and matched with the ultrasonic pulser-receiver instrumentation required considerable ingenuity. To produce ultrasonic energy, a potential difference of several hundred volts was required across the AlN film. Requiring the substrate material to be conductive provided an electrode on one side of the film. The substrate was insulated from the housing (which was

designated as the electrical ground) by a ceramic collar that fit between the substrate and housing. Electrical connection to the other side of the AIN film was achieved by plating the film with platinum and mechanically forcing the platinum-covered surface against the inside of the sensor housing. Attaching the "electrodes" (sensor housing and substrate) to a cable connecting the sensor to the pulser receiver was accomplished by welding the center leads of a metallic-sheathed thermocouple wire to the substrate and clamping the sheath against the sensor housing. The sheath served as the ground wire from the pulser-receiver to the housing. The thermocouple assembly contained an insulating material between the inside "signal" wires and the metallic sheath.

Two approaches may be taken to produce efficient ultrasonic coupling between the sensor and the workpiece in the HIP vessel. The first approach uses the gas in the HIP vessel as the coupling medium while the second uses metals or glasses that soften at 700°C - 800°C as couplants between the sensor and test object. The gas propagation approach proved successful in preliminary tests and was used for all of the prototype sensor testing. Currently, tests are being conducted to assess the viability of using metal films and glass frits as couplants for high temperature testing with the sensor in contact with the workpiece.

Considerable research was done on the propagation of ultrasound in high pressure gases in the 1940's and 1950's (9). Although none of the research extended into the temperature and pressure ranges anticipated in the HIP vessel, enough data existed to imply that the HIP vessel gas could be used as an ultrasonic couplant. Preliminary tests conducted at low temperatures (less than 100°C) over a pressure range from atmosphere to 200 MPa demonstrated that ultrasound between 15 and 25 MHz could be propagated through distances of 100 mm through the gas at pressures above 20 MPa.

SENSOR TESTING RESULTS

The prototype sensor was tested in a furnace in air and at a HIP facility using a research grade HIP vessel capable of applying pressures up to 200 MPa and temperatures greater than 1200°C. A UTEX340 pulser-receiver was used to supply the excitation voltage and receive the signals from the transducer. A 500 MHz, PC-compatible analog-to-digital converter (Signatec, model DA500) was used to convert the voltages from the pulser-receiver to digital data with 8-bit amplitude resolution.

Sensor test in air For the initial check-out tests, the sensor was placed in a clam-shell furnace and the ultrasonic reflection from the back of the substrate was monitored as a function of temperature (the AIN film emits ultrasonic energy from both sides, thus some of the energy propagates into the substrate). The reflection at room temperature was recorded and used for comparison to signals obtained at higher temperatures. During the tests the temperature was limited to 500°C due to the potential for oxidation of the tungsten carbide substrate. Figures 1a and 1b show the reflected ultrasonic signal from the back of the substrate at room temperature and 500°C. No differences in signal amplitude or frequency characteristics is noticeable in the two waveforms.

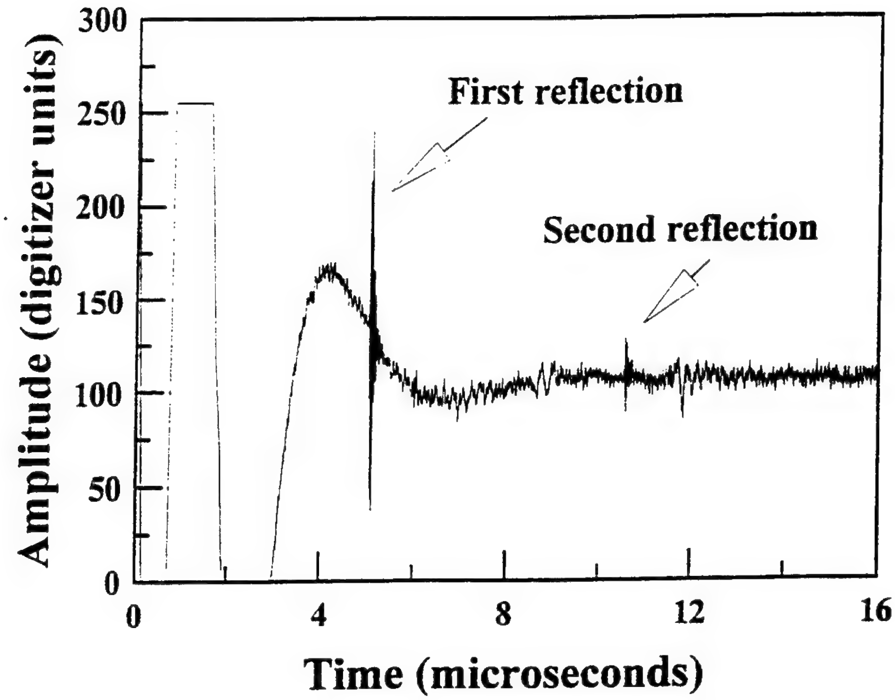

Figure 1a. The ultrasonic signal showing the first and second reflections from the back of the substrate at room temperature.

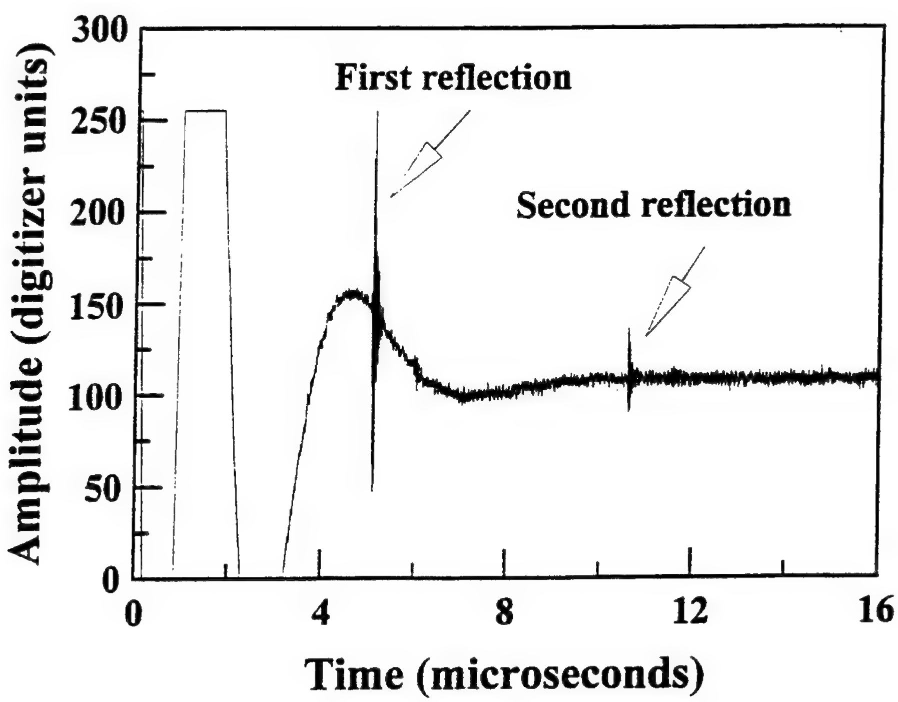

Figure 1b. The ultrasonic signal showing the first and second reflections from the back of the substrate at 500°C. No degradation in signal amplitude is seen when compared to Figure 1a.

Sensor tests in a HIP environment Two HIP vessel tests were conducted using the high temperature sensor. Two different ultrasonic signals were monitored and acquired during the testing. One signal represented the ultrasonic reflection from the back of the substrate. The other signal represented the reflection from a nickel-based superalloy target 33 mm in front of the sensor. The ultrasonic energy reached the target by propagating through the argon gas. During the first test the signal from the back of the substrate was monitored while the pressure and temperature were increased. In the second test both the substrate reflection and a reflection from a target 33 mm away from the sensor were monitored.

During the first test the pressure in the HIP vessel was ramped to 100 MPa while the temperature was held at 65°C. After the pressure reached 100 MPa the temperature was increased to 1000°C at a rate of 10°C per minute. During the temperature ramp the pressure increased to over 155 MPa. Figure 2 shows the amplitude of the ultrasonic reflections from the back of the substrate as a function of temperature. The amplitude varied somewhat as the temperature increased but stayed at approximately 60% full scale (150 digitizer counts) up to 850°C. The last data were recorded at 880°C and the signal was detectable up to 940°C. The temperature in the HIP vessel was increased to 1000°C before cooling down to room temperature. The data recorded at room temperature were obtained with the sensor outside the HIP vessel and connected directly to the pulser-receiver. (This was true for both data points acquired at room temperature - before and after the HIP run. All other data were acquired with the sensor inside the HIP vessel.) The lower signal amplitudes for all data taken above room temperature were due to connecting the sensor to the pulser-receiver using thermocouple wires passing through the HIP vessel wall.

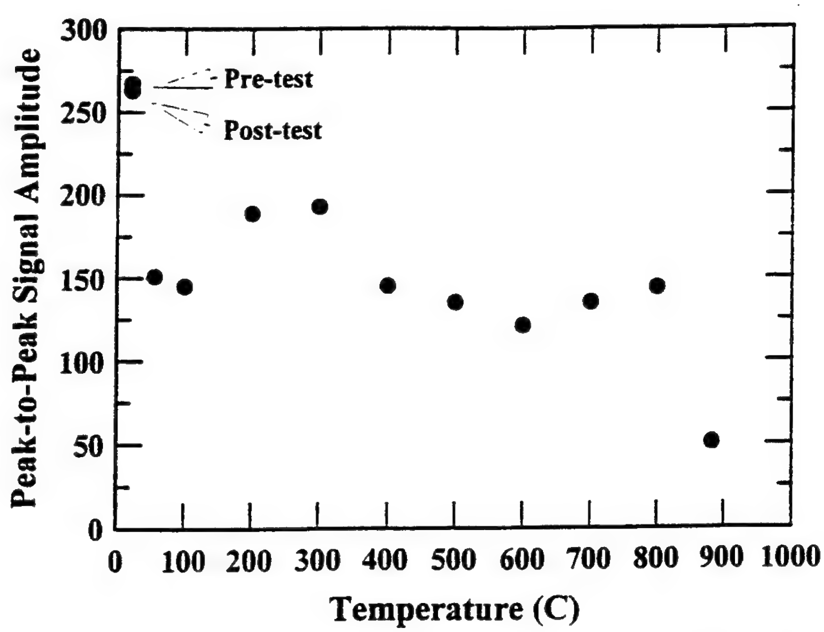

Figure 2. Amplitude data from the reflection from the back of the substrate as a function of HIP temperature. HIP vessel pressure was 150 MPa for all temperatures above room temperature. Pre- and Post-test measurements were performed outside the HIP vessel.

The second HIP run was conducted with the sensor positioned so that the ultrasound emitted from the front face of the AlN propagated through 33 mm of argon gas to a nickel-based superalloy target. The test started by ramping the pressure to 100 MPa at which point a reflection from the target was recorded. The pressure was increased to 155 MPa before beginning the temperature ramp. The temperature in the HIP vessel was increased to 1100°C at a rate of 20°C per minute while holding the pressure constant at ~150 MPa. Ultrasonic data were recorded at approximately 100°C intervals. Figures 3a and 3b show the ultrasonic signal propagating through the argon gas to the target and back at 150°C and 576°C respectively. At 576°C the signal was weaker although still useful for detecting the presence of the target. The ultrasound propagating through argon was detectable up to 676°C, after which it was overwhelmed by electrical noise. The sensor continued to emit ultrasound up to a temperature of 940°C at which point electrical noise overwhelmed the substrate reflection signal making identification of the ultrasonic reflections impossible. The HIP run was continued to 1100°C and then cooled to room temperature. After removing the sensor from the vessel it was determined that the electrical noise arose due to deterioration of the electrical connections to the AlN film. Subsequent testing of the AlN film and substrate, and visual inspection of the housing and internal components revealed no other damage from the hold at 1100°C.

SUMMARY

An ultrasonic transducer was developed that is suitable for use as a displacement sensor in high temperature and high pressure environments. A prototype sensor has been tested to 1100°C and 150 MPa without sustaining damage. The sensor emitted ultrasound at temperatures above 900°C before electrical connection problems occurred. Solutions to the electrical connection problems are being designed. Due to the nature of the piezoelectric properties of the AlN film, it is expected that the film may possibly be used at temperatures exceeding 1200°C. In addition to use as a displacement sensor, work is planned to investigate the possiblity of defect detection, material characterization, and mechanical properties measurements at elevated temperatures.

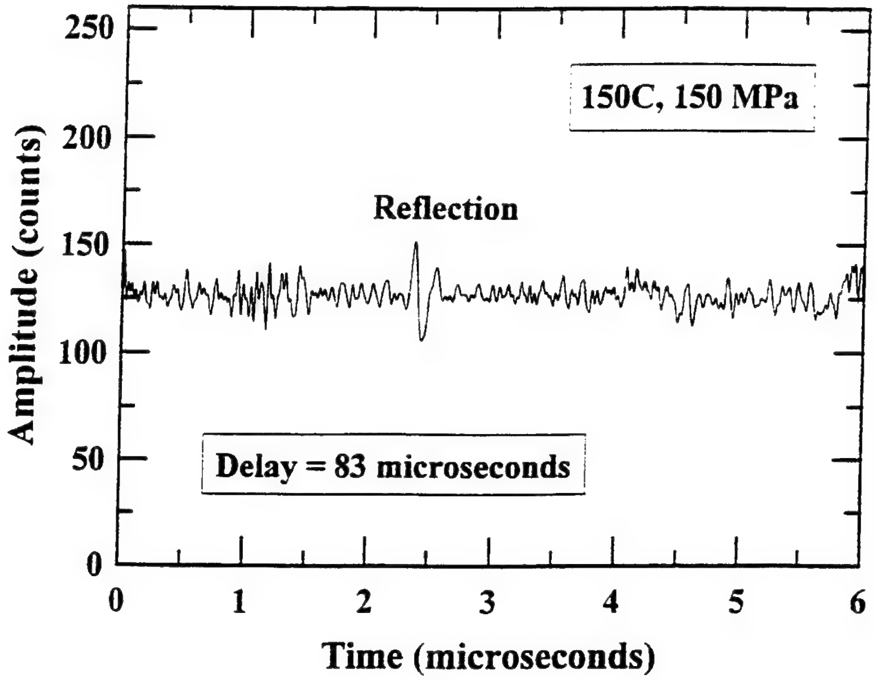

Figure 3a. The reflection from a target 33 mm away from the transducer is shown at 2.5 micro-seconds. HIP temperature and pressure were 150°C and 150 MPa respectively.

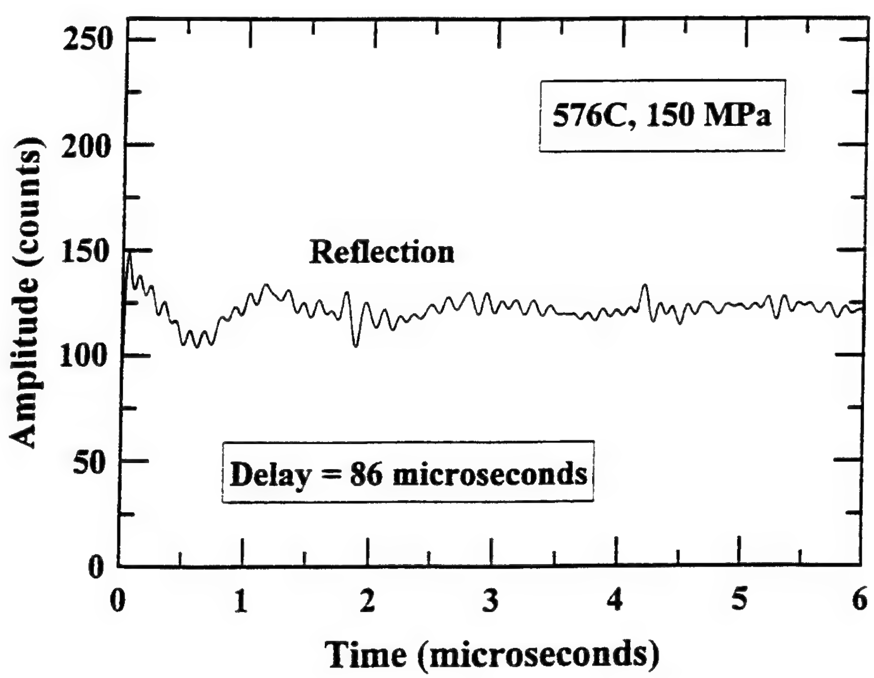

Figure 3b. The reflection from a target 33 mm away from the transducer is shown at approximately 2 micro-seconds. HIP temperature and pressure were 576°C and 150 MPa respectively.

ACKNOWLEDGEMENTS

This work was supported by Air Force contract F33615-95-C-5221 under the administration of Dr. Thomas J. Moran and Dr. Renee M. Kent (Wright Laboratories, Materials Directorate, Metals and Ceramics Division, NDE Branch). The authors acknowledge Dr. N.D. Patel of Fallon Ultrasonics, Inc. for development of the CVD process for the AlN film. The authors wish to acknowledge Dr. Lee Semiatin of the U.S. Air Force (Wright Laboratories, Materials Directorate, Metals and Ceramics Division, Materials Behavior Branch) for supporting the initial development of the AlN material. Appreciation is expressed to Industrial Materials Technology (IMT), Inc. of London, Ohio, for the use of the HIP vessel, and especially to Andrew Clow for assistance in conducting the HIP tests. The authors also acknowledge the technical contributions from the members of the UDRI team: Robert J. Andrews, George A. Hartman, Dr. Prasanna Karpur, William J. Porter, Mark J. Ruddell, Norman D. Schehl, James D. Wolf, Larry D. Sqrow, and Richard A. Grant.

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APPENDIX B

"High-Temperature Ultrasonic Sensor for *In Situ* Monitoring of Hot Isostatic Processing"

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High-Temperature Ultrasonic Sensor for *in Situ* Monitoring of Hot Isostatic Processing

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ABSTRACT

A sensor has been developed and tested that is capable of emitting and receiving ultrasonic energy at temperatures exceeding 900°C (1652°F) and pressures above 150 MPa (22,500 psi). The sensor is based on a unique form of aluminum nitride that retains its piezoelectric properties at high temperatures. The sensor works with standard ultrasonic pulser-receivers and has demonstrated the capability of measuring workpiece deformation during hot isostatic pressing (HIP). Details of the sensor design, performance, and coupling of the ultrasound to the workpiece are described. Ultrasonic data acquired by the sensor, *in situ*, during HIP runs and at elevated tempeatures in air are presented.

Keywords: high temperature ultrasonic sensor, hot isostatic press, HIP, ultrasound, high pressure ultrasonic sensor, aluminum nitride

1. INTRODUCTION

Developments in materials science over the last few years have produced a wide variety of advanced composites and "engineered" materials. As materials become more sophisticated the processes used to produce these materials become more complicated and less "forgiving" than conventional methods of processing. Tight control of the processing parameters is typically required and processing conditions often extend into extreme temperature and/or pressure ranges. While the ability to create and control extreme processing conditions is routinely achieved, sensing mechanisms that provided feedback on the state of the material undergoing processing are scarce.

High temperature structural components, such as structural castings for jet engines, nickel-base superalloy turbine blades, and ceramic and metal matrix composites made from powders are often densified by hot isostatic pressing (HIP). For powder consolidification, the HIP process involves sealing the powder (and reinforcing fibers for composites) within an evacuated "Can" made of a material that softens at high temperatures. A hydrostatic pressure is then applied by pressurizing the inside of the HIP vessel with argon gas (up to 100 MPa). The hydrostatic pressure densifies the powder through the elimination of the porosity between the powder particles

This paper presents details of the development and preliminary testing of an ultrasonic displacement sensor that can be used to monitor materials processing, *in situ*, at temperatures and pressures significantly higher than current ultrasonic displacement sensors. The target processing environment for this sensor is that typically found in a hot isostatic pressure (HIP) vessel, i.e., temperatures exceeding 1000°C (1832°F) and pressures above 138 MPa (20,000 psi).

2.0 CURRENT ELEVATED TEMPERATURE DEFORMATION SENSORS

Dilatometers that can measure the in situ deformation of the HIP work piece during the process cycle have been developed for research applications[1,2,3] but these are limited to simple cylindrical shapes of small size. Dilatometers also are susceptible to malfunctions due to their mechanical nature. Because of the high temperatures and pressures, the only other instrumentation used in industrial HIP applications has been limited to thermocouples that monitor the external

temperature of the HIP work piece. Since it is very difficult to reliably monitor the deformation during the HIP cycle with existing technology, it is typical practice to use the maximum rated pressure of the vessel and to hold at the processing temperature for an extended time to be completely confident that the work piece is fully dense. For some large geometries (such as those developed for NASP structural components) the HIP cycle can last over 24 hours when heat-up and cool-down times are included and cost up to $10,000 per run. In addition, if the HIP "Can" develops a pinhole leak or tear (e.g., due to deformation of the outer wall of the can over a sharp edge) the densification stops due to the equalization of pressure between the exterior and interior of the HIP Can. Currently it is not possible to determine if this has happened until the end of the HIP cycle. Since these leaks often develop during the initial pressurization, the entire run is wasted and must be repeated.

A reliable sensor that could provide information on the actual consolidation of the material would save HIP time and cost. Further, HIP runs could be terminated as soon as consolidation is complete reducing undesired effects due to holding the materials at elevated temperatures for extended periods of time. Recent progress has been made in adapting eddy current sensor technology to HIP vessel consolidation[1]. However, this technology is limited in the range of displacement that can be measured, typically less than 20 mm, and the high cost of the probes.

Standard, room temperature, ultrasonic technology can measure displacements on the order of 0.02 mm (~0.001 inches) over ranges exceeding 100 mm (~4 inches). Successful implementation of the ultrasonic sensor will allow high resolution (0.02 mm), large range (100 mm) displacement monitoring of the consolidation process in a HIP vessel, ultimately producing substantial savings in HIP costs and high reproducibility between components. Considering the extreme environment present in HIP vessels, application of the ultrasonic sensor to other types of high temperature materials processing should be feasible.

At the present, commercially-available ultrasonic transducers are limited to approximately 350°C (662°F) for sustained, *in situ* use because of transducer materials. Extending ultrasonic displacement measurement to an elevated temperature environment creates many problems difficult to overcome. The piezoelectric materials used in typical ultrasonic transducers become inefficient and may lose their piezoelectric properties if temperatures exceed the Curie point of the material. Typical efforts to adapt standard ultrasonic transducers to high temperatures use a "buffer rod" between the transducer and the hot material. For HIP vessel use a buffer rod is unacceptable due to the necessity of breaching the vessel wall.

Several methods of producing ultrasonic energy, other than using piezoelectric materials, have been developed. Electromagnetic acoustic transducers (EMATS) have been used successfully to detect defects and measure mechanical properties of metals at high temperatures[5]. One drawback to using EMATS is the requirement that the target material be electrically conductive. Also, EMATS are generally very inefficient producers of ultrasonic energy so the resulting signal-to-noise ratios are often small.

Recently, laser technology has been used to create and receive ultrasonic energy in a variety of materials[6]. This technique shows considerable promise for producing ultrasonic energy in materials at elevated temperatures, but currently is very expensive and requires the use of moderate power lasers. Also, the application of the laser energy often causes minor damage to the surface of the test object which is undesireable for many materials.

The laser ultrasonic and EMAT techniques, while useful for defect detection and characterization of mechanical properties, are not easily adapted for measuring the displacement of a workpiece in a HIP environment.

3. HIGH TEMPERATURE PIEZOELECTRIC MATERIAL

As early as 1976, aluminum nitride was known to possess piezoelectric properties at high temperatures[7]. Research into the application of aluminum nitride (AlN) to high frequency ultrasonic uses, such as delay lines and other surface acoustic wave devices, was occurring by the mid-1980s since these applications required only very thin AlN films. (The frequency of ultrasound produced by a film is inversely proportional to the film's thickness). In 1990 a chemical vapor deposition (CVD) technique was developed to produce thick layers (100 microns) of AlN in relatively short times (several hours)[8]. The CVD technique made feasible the development of an ultrasonic transducer using AlN that fell in the frequency range of "standard" ultrasonic transducers: 1-50 MHZ. The details of the AlN CVD process, and the structure of the resultant AlN film, are presented in the paper by Patel and Nicholson.

4. HIGH TEMPERATURE, HIGH PRESSURE, ULTRASONIC TRANSDUCER

In 1990 an AlN film was shown to produce ultrasonic energy at temperatures exceeding 1150°C (2102°F) for 24 hours with no degradation in the output of the ultrasonic energy[8]. Recognizing the potential applications in high temperature materials processing for a sensor based on AlN, the University of Dayton Research Institute (UDRI) and the U.S. Air Force began investigations into the requirements for producing a sensor suitable for industrial use. The main tasks of the sensor development program were:

1) Identification of a substrate suitable for deposition of the AlN film
2) Development of a rugged, high temperature sensor housing
3) Development of electrical connection designs that would work at high temperatures
4) Research into methods of coupling the ultrasonic energy to the target object

The remainder of this paper describes the designs resulting from research conducted under each of the four tasks and data from a working prototype sensor under HIP conditions.

4.1 Substrate Development

The requirements for a suitable high-temperature substrate included a thermal coefficient of expansion compatible with the AlN film, high temperature stability, machinability, good adherence of the AlN film, and high electrical conductivity (so that the substrate serves as an electrode for the AlN film.) Several ceramic materials were tried including tungsten carbide, graphite, and silicon carbide. Tungsten carbide and graphite were the final choices due to the adherence of the AlN films and machining/manufacturing considerations. Figures 1 and 2 show the configuration of the substrates and other components used in the prototype sensor.

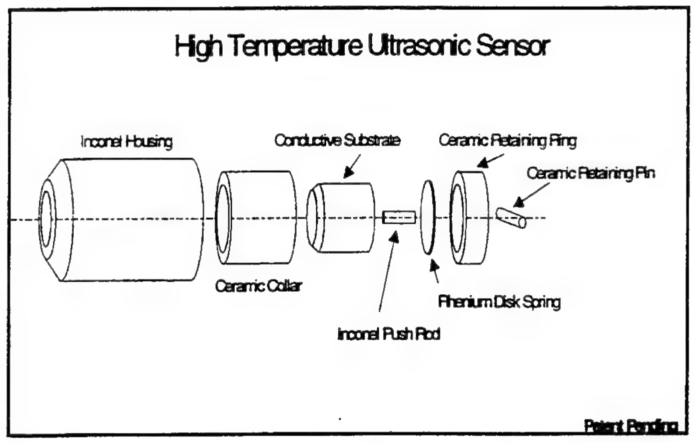

Figure 1 - A schematic drawing showing the primary components of the sensor.

4.2 Sensor Housing

An Inconel® alloy was selected as the housing material because of its high temperature stability and ease of machining characteristics. Careful design resulted in relatively straight-forward machining of the housing at a reasonable cost. The design also allowed off-the-shelf components to be used for electrical connections and insulation purposes. Figure 1 shows the housing design used for the prototype sensor. A rhenium spring / Inconel® push rod assembly was designed to keep the substrate pushed against the front of the housing. Since the thermal expansion of the housing is matched by the expansion of the push rod (both are made of the same Inconel® alloy), the spring force is constant regardless of temperature.

4.3 Electrical Connections

Achieving electrical connections to the AlN film that were stable at high temperatures, and matched with the ultrasonic pulser-receiver instrumentation required considerable research and development. To produce ultrasonic energy, a potential difference of several hundred volts was required across the AlN film. Requiring the substrate material to be conductive provided an electrode on one side of the film. The substrate was insulated from the housing (which was designated as the electrical ground) by a ceramic collar that fit between the substrate and housing. Electrical connection to the other side of the AlN film was achieved by plating the film with platinum and mechanically forcing the platinum-covered surface against the inside of the sensor housing. Attaching the "electrodes" (sensor housing and substrate) to a cable connecting the sensor to the pulser receiver was accomplished by welding the center leads of a metallic-sheathed thermocouple wire to the push-rod and clamping the sheath against the sensor housing. The sheath served as the ground wire from the pulser-receiver to the housing. The thermocouple assembly contained an insulating material between the inside "signal" wires and the metallic sheath. Figure 2 shows a photograph of the disassembled sensor including the sheathed thermocouple wire.

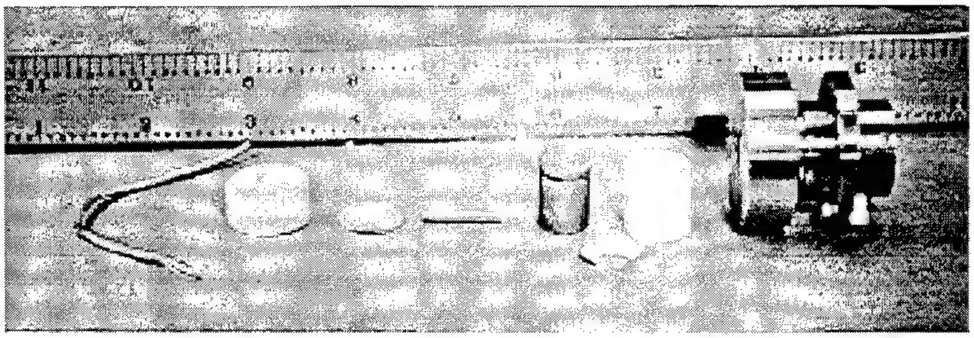

Figure 2 - This photograph shows the components of the sensor. (The ruler has units of inches.)

4.4 High Temperature Ultrasonic Coupling

Two approaches were taken to produce efficient ultrasonic coupling between the sensor and the workpiece in the HIP vessel. The first approach used the gas in the HIP vessel as the coupling medium while the second used metals or glasses that softened at 700°C - 800°C as liquid couplants between the sensor and test object. The gas propagation approach proved successful in preliminary tests and was used for all of the prototype sensor testing. Currently, tests are being conducted to assess the viability of using metal films and glass frits as couplants for high temperature testing with the sensor in contact with the workpiece.

Considerable research was done on the propagation of ultrasound in high pressure gases in the 1940's and 1950's[9]. Although none of the research extended into the temperature and pressure ranges anticipated in the HIP vessel, enough data existed to imply that the HIP vessel gas could be used as an ultrasonic couplant. Preliminary tests conducted at low temperatures (less than 100°C) over a pressure range from atmosphere to 200 MPa (29,200 psi) demonstrated that ultrasound between 15 and 25 MHZ could be propagated through distances of 100 mm through the gas at pressures above 20 MPa (2900 psi).

5. HIP VESSEL TESTING RESULTS

The prototype sensor was tested in a furnace in air and at a HIP facility using a research grade HIP vessel capable of applying pressures up to 200 MPa (29,200 psi) and temperatures greater than 1200°C (2192°F). Figures 3a and 3b show photographs of the furnace, HIP vessel, and ultrasonic data acquisition system. A UTEX340 pulser-receiver was used to supply the excitation voltage and receive the signals from the transducer. A 500 MHZ, PC-compatible analog-to-digital converter (Signatec, model DA500) was used to convert the voltages from the pulser-receiver to digital data with 8-bit amplitude resolution.

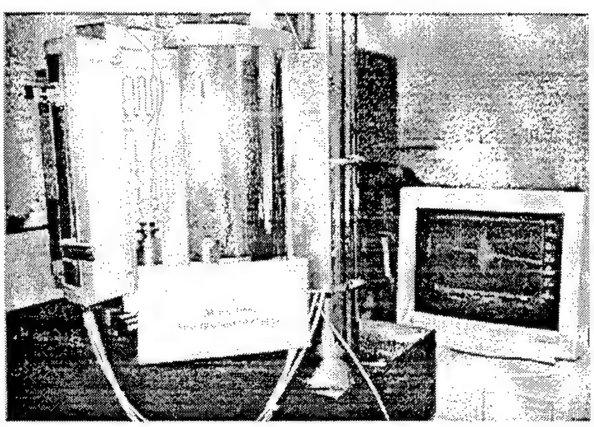

Figure 3a - The sensor is shown inside a clam shell furnace used for tests up to 500 in air°C.

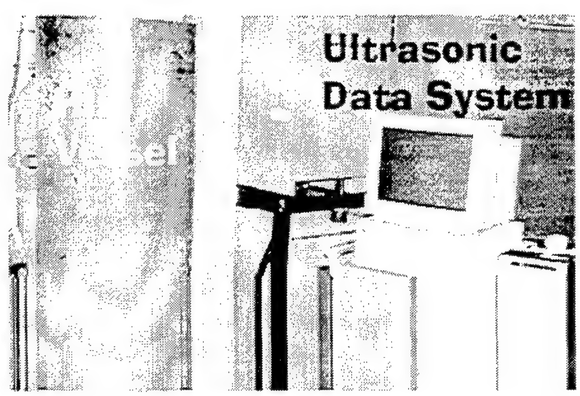

Figure 2b - The ultrasonic data acquisition is shown beside the research HIP vessel used for all HIP tests of the sensor.

5.1 Sensor test in air

For the initial check-out tests, the sensor was placed in a clam-shell furnace and the ultrasonic reflection from the back of the substrate was monitored as a function of temperature (the AlN film emits ultrasonic energy from both sides, thus some of the energy propagates into the substrate). The reflection at room temperature was recorded and used for comparison to signals obtained at higher temperatures. During the tests the temperature was limited to 500°C (932°F) due to the potential for oxidation of the tungsten carbide substrate. Figures 4a and 4b show the reflected ultrasonic signal from the back of the substrate at room temperature and 500°C (932°F). No differences in signal amplitude or frequency characteristics is noticeable in the two waveforms.

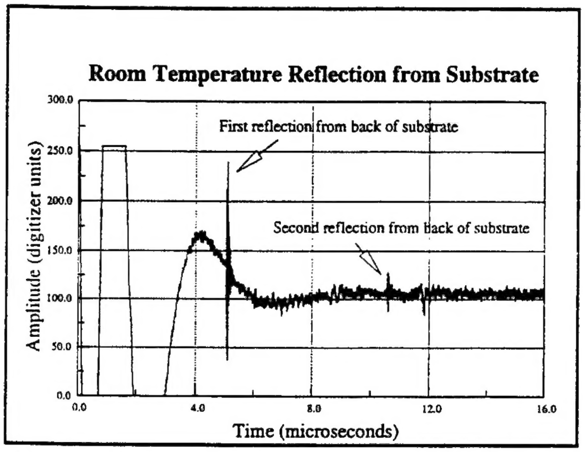

Figure 4a - The ultrasonic signal shows the first and second reflections from the back of the substrate at room temperature.

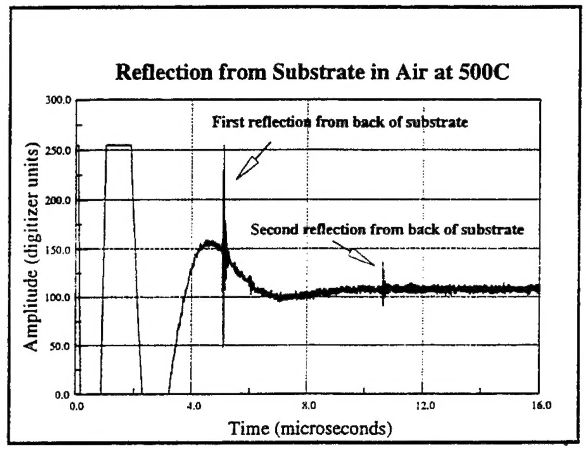

Figure 4b - The signal acquired while the sensor was heated to 500°C in air. No degradation in signal amplitude is seen when compared to the signal shown in Fig. 4a.

5.2 Sensor tests in a HIP environment

Two HIP vessel tests were conducted using the high temperature sensor. Two different ultrasonic signals were monitored and acquired during the testing. One signal represented the ultrasonic reflection from the back of the substrate. The other signal represented the reflection from a nickel-based superalloy target 33 mm in front of the sensor. The ultrasonic energy reached the target by propagating through the argon gas. During the first test the signal from the back of the substrate was monitored while the pressure and temperature were increased. In the second test both the substrate reflection and a reflection from a target 33 mm away from the sensor were monitored.

During the first test the pressure in the HIP vessel was ramped to 150 MPa (~22,000 psi) while the temperature was held at 65°C (150°F). After the pressure reached 100 MPa the temperature was increased to 1000°C at a rate of 10°C per minute. During the temperature ramp the pressure increased to over 155 MPa (22,500 psi). Figure 5 shows

the amplitude of the ultrasonic reflections from the back of the substrate as a function of temperature. The amplitude varied somewhat as the temperature increased but stayed at approximately 60% full scale (150 digitizer counts) up to 850°C (1562°F). The last data were recorded at 880°C (1616°F) and the signal was detectable up to 940°C (1724°F). The temperature in the HIP vessel was increased to 1000°C (1832°F) before cooling down to room temperature. The components of the sensor after removal from the HIP vessel and disassembled, are shown in Figure 6. The components received a coating from the oxidation of a structure used to support the sensor, but were otherwise unaffected by the high temperature/high pressure exposure. The data recorded at room temperature were obtained with the sensor outside the HIP vessel and connected directly to the pulser-receiver. This was true for both data points acquired at room temperature - before and after the HIP run. All other data were acquired with the sensor inside the HIP vessel. The lower signal amplitudes for all data taken above room temperature were due to connecting the sensor to the pulser-receiver using thermocouple wires passing through the HIP vessel wall.

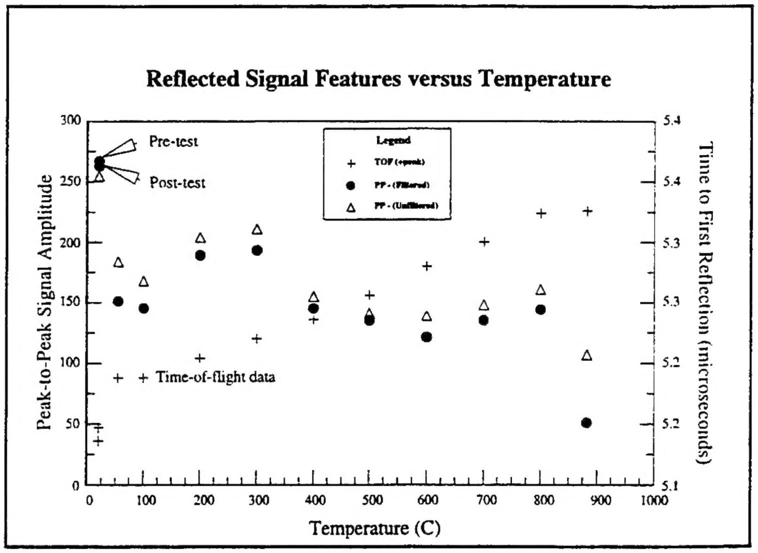

Figure 5 - Amplitude and time-of-flight data from the reflection from the back of the substrate are shown as a function of temperature inside the HIP vessel. The pressure was 150 MPa or greater for all temperatures above room temperature.

The second HIP run was conducted with the sensor positioned so that the ultrasound emitted from the front face of the AlN propagated through 33 mm (1.3 inches) of argon gas to a nickel-based superalloy target. The test started by ramping the pressure to 100 MPa (14,600 psi) at which point a reflection from the target was recorded. The pressure was increased to 155 MPa (22,500 psi) before beginning the temperature ramp. The temperature in the HIP vessel was increased to 1100°C (2012°F) at a rate of 20°C per minute while holding the pressure constant at ~150 MPa. Ultrasonic data were recorded at approximately 100°C intervals. Figures 7a and 7b show the ultrasonic signal propagating through

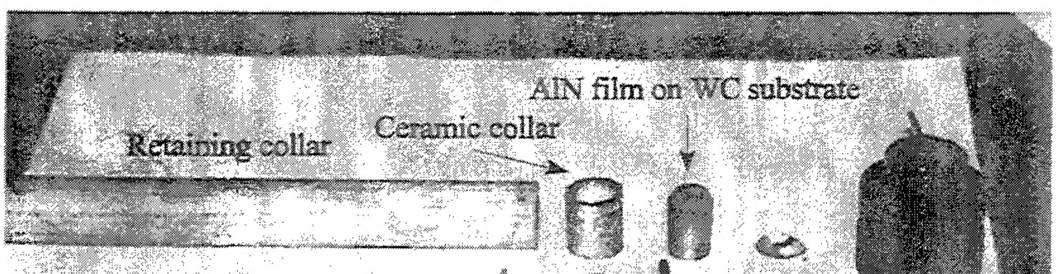

Figure 6 - This photograph was taken of the sensor's components immediately after removal from the HIP vessel. The sensor was heated to 1000°C and subjected to pressures exceeding 155 Mpa during the HIP run. No structural damage was found on any of the sensor's components.

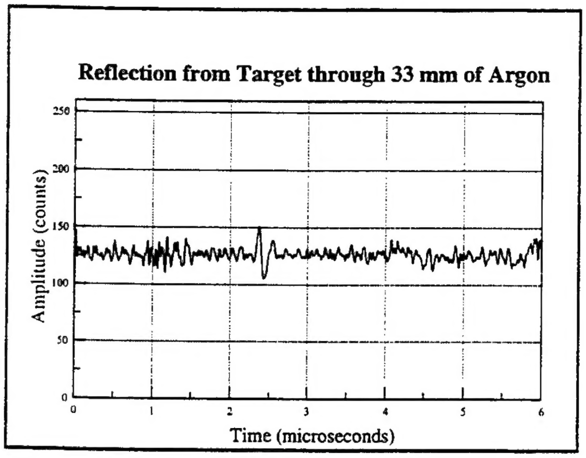

Figure 7a - The reflection from a target 33 mm away from the transducer is shown at 2.5 microseconds. The sensor was in a HIP vessel at 150°C and 150 MPa.

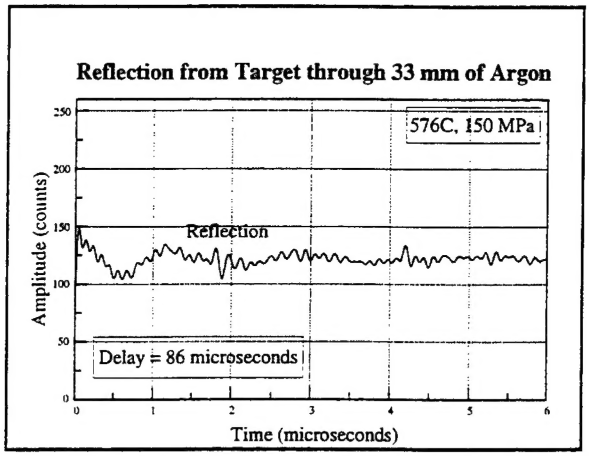

Figure 7b - The reflection from the target is shown at approximately 2 microseconds. The HIP vessel temperature was 576°C and the pressure was 150 MPa.

the argon gas to the target and back at 150°C (302°F) and 576°C (1060°F) respectively. At 576°C the signal was weaker although still useful for detecting the presence of the target. The ultrasound propagating through argon was detectable up to 676°C, after which it was overwhelmed by electrical noise. The sensor continued to emit ultrasound up to a temperature of 940°C (1724°F) at which point electrical noise overwhelmed the substrate reflection signal making identification of the ultrasonic reflections impossible. The HIP run was continued to 1100°C and then cooled to room temperature. After removing the sensor from the vessel it was determined that the electrical noise arose due to deterioration of the electrical connections to the AlN film. Subsequent testing of the AlN film and substrate, and visual inspection of the housing and internal components revealed no other damage from the 1100°C temperature

6. CONCLUSION

An ultrasonic transducer was developed that is suitable for use as a displacement sensor in high temperature and high pressure environments. A prototype sensor has been tested to 1100°C (2012°F) and 150 MPa (22,000 psi) without sustaining damage. The sensor emitted useable ultrasound at temperatures above 900°C (1652°F) before electrical connection problems occurred. Solutions to the electrical connection problems are being designed.

Due to the nature of the piezoelectric properties of the AlN film it is expected that the film can be used at temperatures exceeding 1200°C (2192°F). The current design for the entire sensor also should allow its use at temperatures approaching 1200°C. In addition to use as a displacement sensor, work is planned to investigate the sensor's application to defect detection, material characterization, and mechanical properties measurements at elevated temperatures. The authors welcome communications concerning other potential applications.

7. ACKNOWLEDGEMENTS

This work was supported by Air Force contract F33615-95-C-5221 under the administration of Dr. Thomas J. Moran and Dr. Renee M. Kent (Wright Laboratories, Materials Directorate, Metals and Ceramics Division, NDE Branch). The authors acknowledge the preceding research by Dr. N.D. Patel of Fallon Ultrasonics, Inc. to develop the CVD process for the AlN film. The authors want to acknowledge Dr. Lee Semiatin of the U.S. Air Force (Wright Laboratories, Materials Directorate, Metals and Ceramics Division, Materials Behavior Branch) for supporting the initial development of AlN material. Appreciation is expressed to Industrial Materials Technology (IMT), Inc. in London, Ohio, for the use of the HIP vessel, and Andrew Clow (with IMT) for assistance in conducting the HIP tests. The authors also acknowledge the technical contributions from the members of the UDRI team: Robert J. Andrews, George A. Hartman, Dr. Prasanna Karpur, William J. Porter, Mark J. Ruddell, Norman D. Schehl, James D. Wolf, Larry D. Sqrow, and Richard A. Grant.

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